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SHALE JP-4 ADDITIVE EVALUATION



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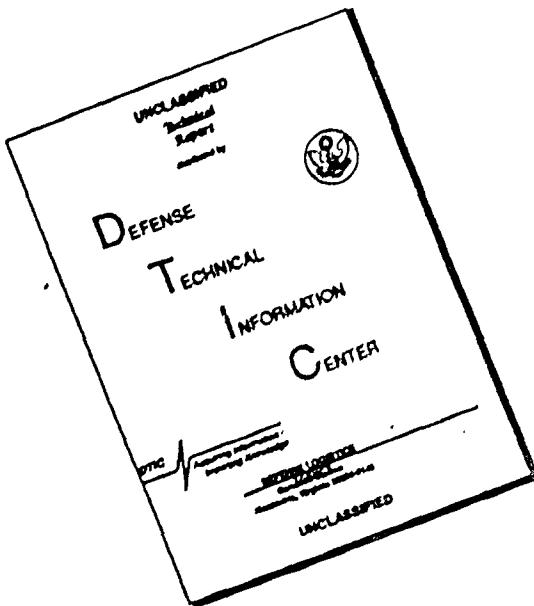
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This technical report has been reviewed and is approved for publication.

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FIELD	GROUP	SUB. GR.							
11	04								
19. ABSTRACT (Continue on reverse if necessary and identify by block number)  <b>A shale JP-4 jet fuel was obtained from the Caribou Refinery of Woods Cross UT as the test fuel. Combinations of additives in varying concentrations were blended to make the test samples. The thermal and storage stability, lubricity, conductivity and water separation characteristics of the samples were studied over 15 months.</b>									
The additives chosen were nine antioxidants, at the minimum and two times the maximum concentration, and four corrosion inhibitors, at the minimum and maximum concentration. The limits were set by the JP-4 specification. Other additives in the test program were Fuel System Icing Inhibitor (FSII), anti-static additive, JFA-5 and metal deactivator. Five-gallon test samples were stored for 15 months: antioxidant samples in a 110°F oven and corrosion inhibitor samples in 70°F - 80°F room storage. Two drums of fuel containing FSII, anti-static additive, and maximum antioxidant and corrosion inhibitor were stored at other conditions, one in cold storage and one at ambient conditions (outdoors). (Key word: (See Reverse))									
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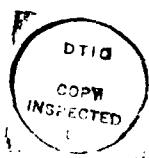
antistatic additive, JFA-5, peroxides, thermal stability, existent gum, water separation, minisonic separometer, naphthalenes, storage stability.

## 19. ABSTRACT

petroleum JP-4 sample was included for comparison purposes. Samples were tested at zero, three, nine and 15 months.

For all test samples, at all test times, the shale fuel met the JP-4 specification requirements for thermal stability (JFTOT), existent gum, particulates and filtration time. Few fuels met the electrical conductivity requirement. This was attributed to the several changes in sample containers. For water separation index, the non-specification minisonic test was used. Using 70 as a minimum requirement, only fuels containing maximum corrosion inhibitor and JFA-5 had failing ratings. All fuels containing maximum corrosion inhibitor did have passing ratings at some time periods.

Other nonspecification tests included peroxides, lubricity and naphthalenes. A minimum amount of antioxidant controlled peroxides to eight parts per million for the 15-month test. Fuel lubricating quality, as determined by the Ball-on-Cylinder Lubricity Evaluato, was poor or marginal (greater than 0.36mm Wear Scar Diameter) for fuels containing the minimum amount of corrosion inhibitor. A maximum amount of corrosion inhibitor generally brought the lubricity up to a "good" rating (less than 0.36mm WSD). No naphthalenes were found in the original fuel.



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## FOREWORD

The Shale JP-4 Additive Evaluation was created as a part of the "Shale Oil Fuel Acceptance Program," established by the Air Force under the program element "Aviation Turbine Fuel Technology," PE63215F. The goal of the acceptance program was to assure the safe use of shale oil derived turbine fuels in operational USAF aircraft and fuel handling systems.

This report describes the additive test fuel origin, additive and fuel amounts, storage conditions, tests performed, and discussion of results and conclusions.

The additive evaluation was carried out by the Fuels Branch of the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was performed under Work Units 24801200 and 30480591. Teresa Boos was the project engineer.

Special thanks go to:

Mr John Yount - SA-ALC/SFTLA, Energy Management Laboratory for performing specification testing,

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## I. INTRODUCTION

In November 1982, the Fuels Branch of the Aero Propulsion Laboratory began a program with the Caribou Four Corners Refinery of Woods Cross UT to refine crude shale oil to meet JP-4 jet fuel requirements under specification MIL-T-5624L. This fuel was used in engine test programs and an additive evaluation program to prove the viability of shale JP-4 as an alternative to petroleum JP-4. This report discusses the additive evaluation program, the test results, and conclusions.

The test was designed to evaluate the effects of various fuel additives on the storage and performance characteristics of shale refined JP-4 fuel. One major portion of the test concentrated on the ability of several specific antioxidants to prevent degradation of the fuel during storage. This degradation was monitored through use of thermal stability breakpoint temperatures, existent gum levels, peroxide levels and particulates. Another portion of the program evaluated fuel lubricity and the effect of additives, including several corrosion inhibitors, on fuel lubricity. In addition, the effect of metal deactivator and JFA-5 on fuel properties was monitored.

The names of the corrosion inhibitors used have been coded to prevent misinterpretation of the data presented. Individual manufacturers may request decoding for their products only. Government agencies may request a complete decoding.

## II. TEST PARAMETERS

### 1. Additives

a. Antioxidants. Seven of the currently approved phenolic antioxidants listed in MIL-T-5624L, one of the earlier approved (and since removed) amine antioxidants, and a resorcinol antioxidant were selected. These are listed in Table 1. The letter codes used to identify the phenolic antioxidants are the same as the JP-4 specification, MIL-T-5624L.

The phenolic antioxidants were chosen based on formulation differences of the primary component; the others in the specification are, for the most part, mixtures containing the above as the major compound. Antioxidant f. is reported to give poor results in preventing peroxidation, and was included for that reason. Antioxidant a. is a very popular, well established product and was selected as the primary antioxidant for the test fuels. The resorcinol antioxidant was added to the test at the three month point at the request of the manufacturer on the strength of data they submitted.

b. Corrosion Inhibitors. Four widely used corrosion inhibitors (MIL-I-25017), identified as CI1 through CI4, were selected for evaluation. CI1 is one of the most widely used corrosion inhibitors and was selected as the "workhorse" corrosion inhibitor for most of the testing, including the antioxidant evaluation.

TABLE 1. ANTIOXIDANTS

A01. N, N'-diisopropyl-p-phenylenediamine

A02. (a) 2,6-di-tert-butyl-4-methylphenol

A03. (b) 6-tert-butyl-2,4-dimethylphenol

A04. (c) 2,6-di-tert-butylphenol

A05. (f) 55% min 6-tert-butyl-2,4-dimethylphenol  
45% max mixture of tert-butylphenols and  
di-tert-butylphenols

A06. (i) 60% min 2,4-di-tert-butylphenol  
40% max mixture of tert-butylphenols

A07. (j) 30% min mixture of 2,3,6-trimethylphenol and  
2,4,6-trimethylphenol  
70% max mixture of dimethylphenols

A08. (k) 65% min mixture of 2,4,5-trisopropylphenol and  
2,4,6-triisopropylphenol  
35% max mixture of other isopropylphenol and biphenyl

A09. 4,6-di-tert-butylresorcinol

c. Conductivity Additives. The two currently approved (MIL-T-5624L) conductivity additives, Shell ASA-3 and DuPont Stadis 450, were evaluated.

d. Fuel System Icing Inhibitor (FSII). The approved FSII, 2-methoxyethanol, MIL-I-27686, was evaluated. Since this program used JP-4 fuel, the higher flash point additive 2-ethoxyethanol (MIL-I-85470) was not included.

e. Metal Deactivator. One of the two approved MIL-T-5624L formulations, N,N'-disalicylidene-1,2-propanediamine, was chosen for evaluation. Based on chemical similarity, it was not felt necessary to evaluate both formulations.

f. JFA-5. This additive is known to improve fuel thermal stability, although it does degrade the water separation characteristics of the fuel. This additive is currently required in JP-TS fuel (MIL-T-25524).

## 2. Additive Amounts.

a. Antioxidants. For Test Series I (Figure 1) all antioxidants were used at the maximum allowable concentration of 8.4 LB/1000 BBL (24.0 mg/liter). For Test Series II (Figure 2), the antioxidant evaluation program, the two test concentrations were the minimum level, 6 LB/1000 BBL (17.1 mg/litre), and twice the maximum level, 16.8 LB/1000 (48.0 mg/liter).

b. Corrosion Inhibitors. For Test Series I both the minimum effective and maximum allowable concentrations were evaluated for effect on fuel lubricity. For Test Series II, a level of 4 lb/1000 BBL was

SHALE ADDITIVE PROGRAM		SAMPLE NUMBER (POSF)																							
		0711 (55 GAL COLD)	0712 (55 GAL AMB)	0751 (CONTROL)	0761 (CONTROL)	0718 (CONTROL)	0718 (CONTROL)	1139	1140	1141	1142	1132	1133	1134	1135	1143	1136	1144	1145	1146	1147	1137	1148	1138	1130
TEST SERIES I		0.10 TO 0.15 VOLUME %																							
ADDITIVES		1 PPM Mix																							
FSII																									
ANTISTATIC																									
A02	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	
A01	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	
CI1	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	
CI2	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	
CI3	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	m	
CI4	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	
METAL DEACTIVATOR																									
JFA-5																									
TESTS																									
SPECIFICATION	x	x	x	x																					
JFTOT																									
PARTICULATES																									
EXISTENT GUM																									
MINISONIC																									
LUBRICITY (BOC)																									
PEROXIDES																									
ELEC CONDUCT																									
NAPHTHALENES	x	x	x	x																					

mx - Maximum allowable by MIL-T-5624L, 8.4 LB/1000 BBL antioxidant, 8 LB/1000 BBL corrosion inhibitor (QPL-25017), 2 LB/1000 BBL MDA, 4 LB/1000 BBL JFA-5 (MIL-T-25524)

mn - Minimum allowable by MIL-T-5624L, 6 LB/1000 BBL antioxidant

% - test performed for that sample

FIGURE 1. TEST SERIES I SAMPLES

SHALE ADDITIVE PROGRAM		SAMPLE NUMBER (POSF)																				
TEST SERIES II		0722 (CONTROL)	0723	1149	0724	1150	0725	1151	0726	1152	0727	1153	0728	1154	0729	1155	0730	1156	1064 (PETROLEUM)	1710	1711	
ADDITIVES																						
FSII		0.10 TO 0.15 VOLUME %																				
ANTISTATIC		1 PPM Mix																				
CI1		4 LB/1000 BBL																				
A02		m	2x																			
A03		m	2x																			
A04			m	2x																		
A05				m	2x																	
A06					m	2x																
A07						m	2x															
A08							m	2x														
A01								m	2x													
A09									m	2x												
TESTS																						
SPECIFICATION		X																				
JFTOT																						
PARTICULATES																						
EXISTENT GUM																						
MINISONIC																						
LUBRICITY (BOC)																						
PEROXIDES																						
ELEC. CONDUCT.																						
NAPHTHALENES		X																				

mn - minimum allowable by MIL-T-5624L, 6 LB/1000 BBL antioxidant

2x - two times the maximum allowable by MIL-T-5624L, 16.8 LB/1000 antioxidant

X - test performed for that sample.

FIGURE 2. TEST SERIES II SAMPLES

chosen, which is the standard amount of inhibitor which most refiners put into their fuel.

c. Conductivity Additive. The two approved additives were used as a mixture at 0.5 ppm each. Field experience shows that this amount usually gives JP-4 the required conductivity.

d. Fuel System Icing Inhibitor (FSII). FSII was added to all samples at 0.10 to 0.15 volume %, the approved level for JP-4. Within this range, the actual amount was not considered critical for test purposes.

e. Metal Deactivator. For the test sequence looking at metal deactivator, the maximum allowable amount of 2 LB/1000 BBL (11.6 mg/liter) was used.

### 3. Test Conditions/Sampling Intervals

Two 55-gallon drums (0711 and 0712) contained fuel with identical additive packages, the former in cold storage (40°F), the latter outside under roof. Except for these two drums, all containers were 5-gallon epoxy lined cans initially filled with 4½ gallons of fuel. The containers were sealed except when removing sample test quantities. Because JP-4 fuel is stored in floating roof or floating pan tanks when in dormant storage, it was not felt necessary to ensure a good supply of air to fuel during storage. Other than sample 0711 and 0712, Series I storage was inside at a controlled/monitored temperature between 70°F-80°F. The Series II test containers were stored at 110°F. The 15-month plan for Series II samples in oven storage was designed to simulate five years of actual storage, using 13 weeks as equivalent to

one year. Fuel samples were evaluated at zero, three, nine and 15 months, with the exception of several samples which were also evaluated at six months for lubricity characterization. The 15-month test was twice interrupted for the Series II 110°F storage samples when oven operation was interrupted. One episode was in December 1984 for seven days and the other was in January 1985 for eight days. The lowest temperature reached on the former incident was 70°F and on the latter 50°F.

#### 4. Fuel Property Tests

Full specification property tests were performed on drum samples (0711 and 0712) for all test intervals and on control fuels for the first test period; in addition, specialized tests such as lubricity, peroxide content, and actual thermal stability breakpoint temperature were performed. The following tests were done on the fuel samples taken at the specified time intervals: thermal stability (JFTOT), particulates, filtration time, existent gum, water separation, lubricity, peroxides, electrical conductivity and naphthalenes. These will be further described in Section IV.

### III. TEST IMPLEMENTATION

#### 1. Origin of Test Fuel

The shale JP-4 test fuel for this test program was obtained from Caribou Four Corners Refinery of Woods Cross, Utah, while the refinery was under subcontract to Geokinetics, Inc. of Salt Lake City, Utah (DoD Contract DLA 600-83-C-5000). The shale crude refined was produced in-situ by Occidental Research Corporation at their Logan Wash Co. facility and by Geokinetics Inc. at Camp Kerogen, UT.

The crude was processed using catalyst and processes licensed from the Union Oil Company. The four refining steps were: distillation, hydrotreating, hydrocracking and product fractionation (Reference 1). Batch II production began July 29, 1983. Non-additive fuel entered the naphtha rundown tank September 26, 1983 (identified as: Tank 528, Batch II, Blend A). All lines had been cleared of additives and the tank cleaned. No clay filtering of the fuel was performed.

The fuel was tested at the Caribou laboratory to determine acceptance as JP-4 with the exception of additive requirements, as specified by MIL-T-5624L. The results are shown in Table 2. Since the fuel did meet specification requirements, concentrated stock solutions of all of the additives were made from a gallon sample of the non-additive fuel.

On September 29, 28 55-gallon drums were filled with 50 gallons each of fuel. The fuel was filtered through a 25 micron fuel filter as it exited the tank. The drums were 16 gauge steel with D.O.T. 17C rating and a double coating of chemically inert IC707 (Rheams epoxy

TABLE 2. RAW SHALE/PETROLEUM FUEL PROPERTIES

PROPERTY	LIMITS	SHALE JP-4 <sup>*</sup>	PETROLEUM JP-4 <sup>**</sup>
Color, Saybolt	1/		+28
Total Acid Number, mg KOH/gm	0.015, max		(0.0008)
Aromatics, vol percent	25.0, max	11.3	(11.9)
Olefins, vol percent	5.0, max	0.5	(0.5)
Sulfur, Mercaptan, wt percent	0.001, max		0.0000
Sulfur, total, wt percent	0.40, max		0.000
Distillation Temperature, °C (D 2887 limits given in parentheses)			
Initial boiling point	1/	52	52
10 percent recovered	T/	83	95
20 percent recovered	T45 (130)	102	111
50 percent recovered	190 (185)	149	142
90 percent recovered	245 (250)	200	179
End point	270 (320)	233	256
Residue, vol percent	1.5, max	0.5	0.5
Loss, vol percent	1.5, max	0.5	0.5
Gravity, deg API or Density, kg/l at 15°C	45.0, min; 57.0, max 0.751, min; 0.802, max	53.3	54.2
Vapor Pressure, psi KPa	2.0, min; 3.0, max 14, min; 21, max	2.4	2.6
Freezing point, °C, (°F)	-58 (-72), max	(-94)	(B-72)
Viscosity, at -20°C, centistokes	42.8 (18,400), min	18,736	(1.8)
Net heat of combustion, MJ/kg (Btu/lb)	13.6, min 20.0, min	14.5	43.5
Hydrogen content, wt percent or Smoke point, mm		14.3	14.3
Copper strip corrosion, 2 hr at 100°C (212°F)	1b, max	1A	1A
Thermal stability			
Change in pressure drop, mm Hg heater tube deposit visual rating	25, max ≤3	0 1	(0) (1) (2)
TDR rating			

TABLE 2. RAW SHALE/PETROLEUM FUEL PROPERTIES (CON'T)

PROPERTY	LIMITS	SHALE JP-4*	PETROLEUM JP-4**
Existent gum, mg/100 ml	7.0, max	1.0	
Filtration time, min	15, max	3	
Particulate matter, mg/liter	1.0, max	0.5	
Water reaction			
Interface rating	1b		
Water separation index, modified	2/		
Fuel system icing inhibitor, vol percent	0.10, min; 0.15, max		
Fuel electrical conductivity, p/S <sub>m</sub>	200, min; 600 max		
Corrosion Inhibitor, 1b/Mbbl (DCI-4A)		3.1	

\* Caribou Refinery Data - some tests not available

\*\* Hill AFB Data

( ) SFTLA, Wright-Patterson AFB

1/ To be reported - not limited

2/ The minimum water separation index, modified, rating for JP-4 shall be 85 with all additives except corrosion inhibitor and electrical conductivity additives present, or 70 with all additives except for the electrical conductivity additives.

coating #973). All drums were checked to insure dryness, but none were pre-rinsed with shale fuel.

Fuel was added to drums through the bunghole while a measured volume of additive solution was added through the vent hole. Table 3 lists the drum identification numbers and concentration of additives they contained. These drums were shipped to Wright-Patterson AFB on September 30.

The 28 drums arrived at Wright-Patterson October 5, 1983. They were tumbled on a drum shaker for 15 minutes each to insure that all additives were thoroughly mixed with the fuel. Fuel conductivity was then measured and recorded, as was the fuel temperature. A concentrated solution of ASA-3 and Stadis 450 in toluene was added to all fuels which registered lower than 200 pS/m (picosiemens per meter or Conductivity Units, CU). These were 0713, 0727 and 0728. The aliquot of solution added to a drum increased the concentration of anti-static additive by 0.5 ppm. After the addition, the drum was tumbled for 15 minutes and a new reading taken.

During this same time period, a petroleum JP-4 (drum 1064) was obtained from Hill AFB, Utah, for use in the test as a reference fuel. It also was shipped in an epoxy lined drum. Specification test results are shown in Table 2.

## 2. Sample Preparation

A list of the sample numbers and their additive packages are listed in Figures 1 and 2. The test samples which could be made from the original drummed fuel, that is those which contained the same

TABLE 3. DRUM SAMPLE ADDITIVE CONTENT

All units for DCI-4A and antioxidants are pounds per thousand barrels.

## SHALE ADDITIVE PROGRAM

AF FORM 55877 313

## GENERAL PURPOSE (10%--X 8%)

combination and concentration of additives, were drawn (Appendix A). The fuel remaining in the drums was mixed with additive solutions in order to make the rest of the test samples. One exception to this was the 4-6 di-tertiary-butylresorcinal samples, 1710 and 1711, which was formulated using 0722 drummed fuel at the three-month point. The solutions were concentrated quantities of additives in toluene. They were made up the week the drums arrived. Drums were tumbled for one half hour, then sampled.

All fuel test samples (except 0711 and 0712 which were 55-gallon drums) were stored in two five-gallon epoxy lined cans. Each can was rinsed with the fuel to be stored in that can and then filled to approximately four and one half gallons.

### 3. Test Fuel Sampling

Sampling for each test period followed this procedure:

1. Shake can
2. Rinse two one-gallon cans with test fuel
3. Fill two one-gallon cans plus one 250ml glass bottle with test fuel

One one-gallon can was given to SA-ALC/SFTLA, the Energy Management Laboratory, Wright-Patterson AFB, for the particulate/filtration time test. SFTLA also received the 250ml bottle for the remainder of their tests: water separation and existent gum. The Aero Propulsion Laboratory's (APL) Fuels Branch received the other one gallon can for their tests: lubricity, thermal stability, peroxides and electrical

conductivity. For room storage test samples (Series I), some APL tests were done directly from the five gallon can storage containers.

To differentiate between the first and second five gallon can of each sample, the cans were labeled "A" and "B" (or "C" and "D" for duplicate samples). All zero-, three-, and six-month tests were done on samples drawn from the "A" and "C" cans. All nine and fifteen month samples were done on samples drawn from "B" and "D" cans, except the following:

0722	0725	1139
0730	0727	0761
0718	0761	0718

For these samples, in the fifteen-month tests, "B" and "D" cans were used in SFTLA tests while "A" and "C" were used for APL tests. One exception to this was 0718B, which was also used for the particulate/filtration time test.

#### IV. TEST RESULTS AND ANALYSIS

##### 1. Thermal Stability (JFTOT).

Thermal stability of the test fuels was measured using the Jet Fuel Thermal Oxidation Tester (JFTOT) in accordance with the ASTM D3241 procedure. All samples were tested at the breakpoint temperature of the original fuel, i.e., the breakpoint of 0761 (310°C) for Series I fuels and of 0722 (320°C) for Series II fuels. If a sample passed at the original temperature, the test was complete. If the first test was a failure, the test was rerun at 20°C below the breakpoint temperature. If the second test was a failure, the test was rerun at 40°C below the original breakpoint temperature. A pass at 260°C, a visual rating of less than three and a pressure drop of less than 25 mm Hg, is the JP-4 specification requirement. The tube deposit ratings (TDR) for all tests are reported for a further indication of fuel thermal stability. Though the JP-4 specification does not have a TDR limit, both JP-7 and JP-TS specifications require that the tube rating not exceed 12.

Series I test samples were stored for 15 months at room temperature while Series II samples were in oven storage at 110°F for 15 months, simulating five years of storage at room temperature. Thus, a good JFTOT performance in Series I was not as severe a test of a sample as compared to Series II, and samples not performing well in Series I were considered to have very poor thermal stability.

The Series I fuels that failed at the original test temperature are listed in Table 4. Fuels listed here contained the maximum allowable amount of both antioxidant and corrosion inhibitor. All of the Series I

TABLE 4. SERIES I - JFTOT FAILS AT ORIGINAL TEST TEMPERATURE

FUEL CODE	STORAGE	FSII	ANTI-STATIC	CORROSION INHIBITOR		OTHER LB/1000 BBL
				LB/1000 BBL	LB/1000 BBL	
1130 A/B	ROOM	YES	NO	8.4 A02	8	C11
1131 A/B	ROOM	YES	NO	8.4 A01	8	C11
1132 A/B	ROOM	YES	YES	8.4 A01	8	C11
1133 A/B	ROOM	YES	YES	8.4 A01	8	C12
1134 A/B	ROOM	YES	YES	8.4 A01	8	C13
1138 A/B	ROOM	YES	YES	8.4 A01	8	C11
1141 A/B	ROOM	YES	YES	8.4 A02	8	C13

## JFTOT RATINGS

## MONTHS

FUEL CODE	TEMP °C	VIS CODE	ΔTDR mmHg	0		VIS CODE	ΔTDR mmHg	3		VIS CODE	ΔTDR mmHg	9		VIS CODE	ΔTDR mmHg	15	
				ΔP mmHg	ΔP mmHg												
1130 A/B	310	2	5	0.5	4	13	0.3	1	5.5	0.1	3	11.5	0.1	1	1.5	0.2	0
1131 A/B	310	2+	12	0	3+	20.5	5	1	7	0	2	8	0	2	8	0	0
1132 A/B	310	2	8.5	0	4	10	0	2	15	0	4+	9	0	1	4	0	0
1133 A/B	310	4	7.5	0	4	23.5	0	1	2	0	2	4	0	2	4	0	0
1134 A/B	310	1	1	0	1	1	0	1	1	0	1	1.5	0	1	1.5	0	0
1138 A/B	310	1	2.5	0	3	5	0.1	2	5	*	1	1.5	0	1	1.5	0	0
1141 A/B	310	1	4	0.3	3	3	0.2	1	1.5	*	1	2	0	1	2	0	0
	290	1	1	0	1	1.5	0	1	1	*	1	1.5	0	1	1.5	0	0
	290	1	2.5	0	1	4	0.1	1	1	0	1	1.5	0	1	1.5	0	0
	290	1	4	0.3	3	3	0.2	1	1.5	*	1	2	0	1	2	0	0
	290	1	1	0	1	1	0	1	1	*	1	1.5	0	1	1.5	0	0
	290	1	2.5	0	1	13	0.2	1	11.5	*	3	14	0.2	1	2	0	0

\* Pressure Transducer Non-Operative

fuels with the combination of maximum allowable A01 and maximum allowable corrosion inhibitor appear in the table with exception of a sample containing CI4 (1135) and a sample containing metal deactivator (1146). CI4 seems to aid thermal stability, as does metal deactivator, when combined with the maximum concentration of A01. One Series I sample (1137) containing maximum A01 and a minimum amount of corrosion inhibitor, CI2, did very well.

In Series I the CI3 corrosion inhibitor at maximum concentration degraded thermal stability. Its combination with either maximum A01 (1134) or maximum A02 (1141) caused samples to fail at the original test temperature. All Series I samples containing the minimum allowable concentration of corrosion inhibitor showed good thermal stability.

An unlikely sample for poor thermal stability was 1130 (Table 4), which contained the maximum concentration of A02. Its poor performance is attributed to fuel degradation during shipment from Caribou Refinery, since at that time the fuel from which this sample was made contained no additives. Comparatively, samples containing a similar additive package (0711, 0712, and 1139) showed no thermal stability problem.

The JFA-5 sample (1138), failed at one time period. Overall, JFA-5 was effective in improving thermal stability, as evidenced when sample 1138 is compared to sample 1131, which contained the same additive package with the exception of JFA-5. The JFA-5 sample had lower visual codes, tube deposit ratings, and pressure drop. A sample similar to 1138, containing JFA-5 and A02 rather than the A01 antioxidant (1148), did not fail at the original test temperature.

Metal Deactivator (MDA) improved the thermal stability of the shale fuels. Comparing a sample containing FSII, anti-static additive, maximum CI1 and A02 (1139) to a like sample with MDA (1143), the MDA sample had all ones for visual code with low TDR and  $\Delta P$ , while sample 1139 had visual ratings of 1 and 2, with TDRs of 14 and 10, respectively. Likewise for the A01 samples, the MDA sample (1136) had a visual code of 1 with low TDR and  $\Delta P$ , while the same sample without MDA (1132) failed the visual rating at 3 and 15 months and registered a 15 TDR at nine months.

In test Series II, antioxidants were evaluated. The worst performing fuels were those that failed in the 15th month (Table 5). Some samples failed earlier in the test program, but not at the 15th month, so that results for those samples showed test inconsistencies rather than fuel degradation. Samples containing no antioxidant (0722) did not do well, though they were still passing thermal stability at 280°C, which is higher than the JP-4 specification. Two fuels containing antioxidant A03 and A08 (1150 and 1155) at the 16.8 LB/1000 BBL concentration, failed at fifteen months, while their 6 LB/1000 BBL counterparts (0724 and 0729, respectively) passed at the original test temperature. This suggests that a high concentration of some antioxidants may in fact be detrimental to fuel thermal stability. Samples containing A01 in both the maximum and minimum allowable concentration (0730 and 1156) did not do well, failing at the original test temperature for all test periods. However, these samples did better than samples containing no antioxidant, by passing at 300°C.

TABLE 5. SERIES II - JFTOT FAILS AT 15 MONTHS

FUEL CODE	STORAGE	FSII	ANTI-STATIC	CORROSION			OTHER LB/1000 BBL
				ANTIOXIDANT LB/1000 BBL	INHIBITOR LB/1000 BBL		
0722 A/B	OVEN	YES	YES	NO	NO	4 CII	
0722 C/D	OVEN	YES	YES	NO	NO	4 CII	
0730 A/B	OVEN	YES	YES	6 A01	4 CII		
1150 A/B	OVEN	YES	YES	16.8 A03	4 CII		
1155 A/B	OVEN	YES	YES	16.8 A08	4 CII		
1156 A/B	OVEN	YES	YES	16.8 A01	4 CII		

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FUEL CODE	TEMP °C	VTS CODE	0 ΔTDR mmHg	3 ΔP mmHg	VTS CODE	9 ΔTDR mmHg	ΔP mmHg	15	
								VTS CODE	ΔTDR mmHg
0722 A/B	320	1	8.5	0	2	19.5	0	2	4.5
0722 C/D	300	--	--	--	--	--	--	--	--
0730 A/B	320	2	10	3	2	10.5	0	1	4
1150 A/B	320	4	15.8	0.1	4+	31	0	3	12
1155 A/B	320	1	1.5	0.3	1	2	0	1	2
1156 A/B	320	2	3.9	1	4+	31	0.1	1	5
	300	--	--	--	--	--	--	--	--
	300	--	--	--	--	--	--	--	--
	300	--	--	--	--	--	--	--	--
	300	--	--	--	--	--	--	--	--

The Series II fuels which performed best were those that passed at all test periods (Table 6). From these results, the minimum 6 LB/1000 BBL of A02, A04, A08, or A09 is adequate to protect fuel thermal stability. The fuels containing 16.8 LB/1000 of these additives did well also, passing at the fifteen month test, except A08 (1155).

## 2. Particulates/Filtration Time

The ASTM D2276 method, "Particulate Contamination in Aviation Turbine Fuels," was used to determine particulates, using a one gallon sample as prescribed by the JP-4 specification and following the method outlined there for determining filtration time. With two exceptions, all sample results were well within the JP-4 specification limits of 1 mg/liter particulates and 15 minutes filtration time for all test periods.

The two exceptions were 0722 and 1149. The 0722 B and D samples had particulates of 0.5 and 1.0 mg per liter, respectively. These two samples contained no antioxidant, and thus some particulate formation was expected. These fuels still met the JP-4 specification requirements. The 1149 sample had a 17.2 mg per liter particulate level at the zero month test. This result was caused by a piece of rubber-like substance found in the sample and was surmised to have come from the can lining.

## 3. Existent Gum

Existent gum content was determined by ASTM D381, "Existent Gum in Fuels by Jet Evaporation," as required by the JP-4 specification. All

TABLE 6. SERIES II - NO JFTOT FAILS AT ORIGINAL TEMPERATURE

FUEL CODE	STORAGE	FSII	ANTI-STATIC	ANTI-OXIDANT		CORROSION INHIBITOR		OTHER LB/1000 BBL
				LB/1000 BBL	LB/1000 BBL	LB/1000 BBL	LB/1000 BBL	
0723 A/B	OVEN	YES	YES	6 A02		4 C11		
0725 A/B	OVEN	YES	YES	6 A04		4 C11		
0729 A/B	OVEN	YES	YES	6 A08		4 C11		
1153 A/B	OVEN	YES	YES	16.8 A06		4 C11		
1710 A/B	OVEN	YES	YES	6 A09		4 C11		
1711	OVEN	YES	YES	16.8 A09		4 C11		

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FUEL CODE	TEMP °C	0		3		9		15	
		VTS CODE	ΔTDR mmHg						
0723 A/B	320	2	24.2	0.3	2	20	0.1	1	3
0725 A/B	320	2+	20	2	1	12	0	2	11
0729 A/B	320	1	3.5	0.2	2	18	0	1	11
1153 A/B	320	2	10.5	0.5	2	20	0.9	1	1
1710 A/B	320	--	--	--	1	3	0.4	1	7
1711 A/B	320	--	--	--	1	7.5	1	1	3

\* Pressure Transducer Non-Operative

samples were well under the JP-4 specification limit maximum of 7 mg/100 ml for all time periods.

The highest gum content was in a sample containing no antioxidant (0722 C and D), which registered 3.4 mg gum per 100 ml of fuel. A duplicate sample (0722 A and B) contained no gum at the fifteen month test. One other sample, containing a maximum allowable amount of A01 and a maximum allowable amount of CI3 (1134), had 3.2 mg/100 ml gum. This sample was at room temperature and should not have experienced any gum formation. The result is considered insignificant considering the results described above for duplicate samples of 0722. 1134 A and B had good results until the 15th month. A sample similar to 1134, 1141, containing maximum CI3 with antioxidant A02 rather than A01, had no gum at 15 months.

#### 4. Peroxides

Peroxide content of the test samples was determined by ASTM D3703, "Peroxide Number of Aviation Turbine Fuels." The MIL-T-5624L specification does not state a peroxide limit for JP-4, but sets 1 meq/1000g (8 ppm) as the maximum allowable for JP-5. This is the standard for comparison of these test results. The fuels tested for peroxides were all of those in Series II (oven storage) and selected samples from Series I. The Series II fuel samples contained a variety of antioxidants at the minimum and two times the maximum allowable concentration.

Over all time periods, the shale fuel performed very well, with 19 of 28 samples having less than one part per million peroxide by the

fifteenth month. At fifteen months, only two fuels (0722 A/B, C/D) were above the 8 ppm limit; they contained no antioxidant.

The 0722 fuels A/B and C/D were duplicates. A similar sample, containing no antioxidant, was 0761 A/B and C/D. Sample 0722 A/B and C/D (oven storage) showed 1000 ppm peroxides. Sample 0761 (room temperature) showed zero ppm peroxide by the end of the test. This indicates that the shale fuel with no antioxidant can be stable (no peroxides) at room temperature for at least 15 months (as indicated by 0761) and up to three years\* (as indicated by 2 ppm peroxide for 0722 C/D at nine months).

Since all of the antioxidants kept the peroxide level to less than eight ppm, the best antioxidant was determined as the one which kept the peroxide level at zero through the fifteenth month with the lowest concentration of additive. Three additives fell into this category: A02, A05 and A01. Barely distinguishable from these are those antioxidants which controlled peroxides to less than one ppm at the minimum concentration: A03 (0724), A04 (0725), and A06 (0727). Though these results may seem insignificant, they occurred at the 9 and 15-month test period, and are seen as an indication of an upward trend in the amount of peroxides. Of samples containing two times the maximum amount of these antioxidants 1150, 1151, and 1153, respectively, 1150 contained no peroxides at 15 months, while 1151 and 1153 contained some peroxides, still less than one part per million.

Three of the nine antioxidants tested, A07 (0728, 1154), A08 (0729, 1155), A09 (1710, 1711), as listed Table 7, did not keep peroxides to

\* Using 1 wk oven = 4 wks ambient equivalent

below 1 ppm. As seen in this table, even two times the maximum concentration of these three antioxidants did not prevent peroxides from forming. Comparing their structure with the other antioxidants (Table 7A), the phenolic antioxidants with tertiary butyl side chains prevented peroxide formation better than those with isopropyl chains, such as A07 and A08. Also, the resorcinol antioxidant, A)9, did not perform as well as the tertiary butyl phenolic antioxidants.

It should be noted here that the A09, added to the program at three months, may have performed better if it had been added to fuel at the zero month. However, the 0722 fuel drum from which these samples were made had been in cold storage. In addition, the 0722 test samples, which were at 110°F, showed less than one part per million peroxide at three months.

For most fuels containing peroxides, the amount progressively increased to the fifteenth month, so that fuels with zero ppm peroxides contained the best antioxidants. For all antioxidants at all test periods, a minimum concentration of antioxidant did as well as two times the maximum concentration.

##### 5. Water Separation

Water Separation was measured using the ASTM D3602 procedure for a Minisonic Separometer, "Water Separation Characteristics of Aviation Turbine Fuels." All test fuels underwent a Minisonic test at each of the four test times. The JP-4 specification requires a minimum Water Separation Index Modified (ASTM D2550) of 85 with all additives except

TABLE 7. FUELS WITH PEROXIDES ONE THROUGH EIGHT (ppm) AT 15 MONTHS

FUEL CODE	STORAGE	ICING INHIBITOR	ANTI-STATIC	ANTIOXIDANT LB/1000 BBL	CORROSION INHIBITOR LB/1000 BBL	PEROXIDES (ppm)			
						0	3	9	15
0728	OVEN	YES	YES	6 A07	4 C11	0	0	0.160	1.442
0729	OVEN	YES	YES	6 A08	4 C11	0	0.087	0	1.420
1710	OVEN	YES	YES	6 A09	4 C11	-	0	0.640	5.560
1154	OVEN	YES	YES	16.8 A07	4 C11	0	0	1.740	4.420
1155	OVEN	YES	YES	16.8 A08	4 C11	0	0	0.480	1.553
1711	OVEN	YES	YES	16.8 A09	4 C11	0	0	3.400	1.830
								3.000	5.710
						-	0	0.880	2.830

TABLE 7A. RELATIONSHIP OF ANTIOXIDANT STRUCTURES TO PEROXIDES

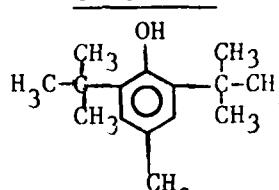
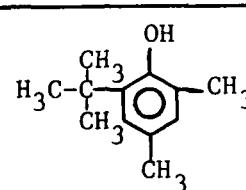
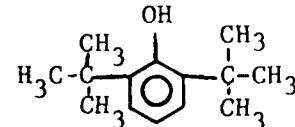
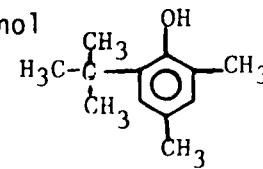
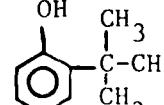
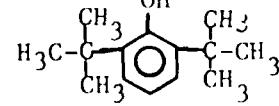
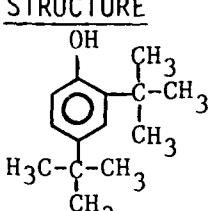
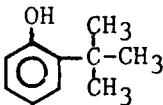
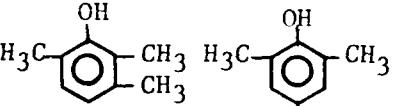
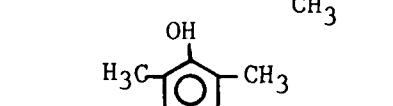
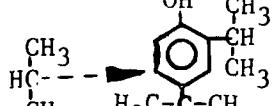
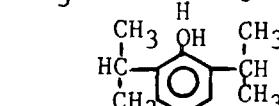
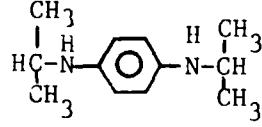
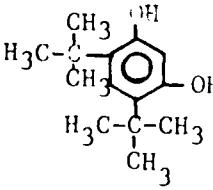
<u>ANTIOXIDANTS</u>	<u>STRUCTURE</u>	<u>PEROXIDES AT 15 MONTHS, ppm</u>	
A02 2,6-di-tert-butyl-4-methylphenol		0723-mn	0
		1149-2xmx	0
A03 6-tert-butyl-2,4-dimethylphenol		0724-mn	0.415
		1150-2xmx	0
A04 2,6 di-tert-butylphenol		0725-mn	0.520
		1151-2xmx	0.525
A05 6-tert-butyl-2,4-dimethylphenol tert-butylphenols di-tert-butylphenols		0726-mn	0
		1152-2xmx	0
			

TABLE 7A. RELATIONSHIP OF ANTIOXIDANT STRUCTURES TO PEROXIDES (CON'T)

<u>ANTIOXIDANTS</u>	<u>STRUCTURE</u>	<u>PEROXIDES AT 15 MONTHS, ppm</u>	
A06 2,4-di-tert-butylphenols tert-butylphenols	 	0727-mn	0.526
		1153-2xmx	0.701
A07 2,3,6-trimethylphenol 2,4,6-trimethylphenol dimethylphenols	  	0728-mn	1.400
		1154-2xmx	1.708
A08 2,4,5 triisopropylphenol 2,4,6 triisopropylphenol	 	0729-mn	4.990
		1155-2xmx	6.151
A01 N-N'-diisopropyl-p-phenylenediamine		0730-mn	0
		1156-2xmx	0.841
A09 4,6-di-tert-butyl resorcinol		1710-mn	1.646
		1711-2xmx	2.306

corrosion inhibitor and electrical conductivity additives present, or 70 with all additives except for the conductivity additives. Though the specification calls for a different test than was used in this program, the 70 index was used to relatively evaluate the fuels. With the exception of two fuels (1130 and 1131), all fuels contained an anti-static additive, and, with the exception of one (0761), contained corrosion inhibitor, so that results were expected to fluctuate (Reference 3).

Ten fuels had at least one failure (less than 70 rating), of which seven had more than one failure. The seven fuels which did not perform well are listed in Table 8. All of these contained the maximum allowable amount of corrosion inhibitor and the maximum allowable amount of antioxidant, with the exception of 1711.

For the first four fuels, corrosion inhibitor at the maximum concentration (8 LB/1000 BBL) was the cause of the low water separation as measured by the minisonic test, since none of the Series II samples (except 1711, which contained 4 LB/1000 BBL corrosion inhibitor with two times the maximum amount of antioxidant) failed the test. These failures are attributed to poor test precision, since these fuels did have high ratings at some time periods. All test fuels containing the maximum amount of corrosion inhibitor failed the test at least once except one which contained the maximum CI2 with the maximum amount of antioxidant A01 (1140).

In general, a maximum concentration of antioxidant did not affect water separation as measured by the Minisonic. An exception was A09 (1711), which adversely affected water separation when present at two times the maximum allowable concentration.

TABLE 8. FUELS FAILING WATER SEPARATION TEST

FUEL CODE	STORAGE	FSII	ANTI-STATIC	ANTIOXIDANT LB/1000 BBL	CORROSION INHIBITOR LB/1000 BBL	OTHER LB/1000 BBL	WATER SEPARATION INDEX MONTHS			
							0	3	9	15
0712	AMBIENT	YES	YES	8.4 A02	8 C11		66	85	70	66
1134 A/B	ROOM	YES	YES	8.4 A01	8 C13		63	56	44	67
1135 A/B	ROOM	YES	YES	8.4 A01	8 C14		71	61	58	67
1143 A/B	ROOM	YES	YES	8.4 A02	8 C11	2 MDA	85	91	57	67
1138 A/B	ROOM	YES	YES	8.4 A01	8 C11	4 JFA-5	39	57	41	56
1148 A/B	ROOM	YES	YES	8.4 A02	8 C11	4 JFA-5	49	55	40	61
1711 A/B	OVEN	YES	YES	16.8 A09	4 C11	--	--	65	57	57

Further, JFA-5 affected water separation adversely. The two test samples which contained JFA-5, (1138 and 1148) had the lowest ratings for all of the fuels tested and failed at all time periods.

For most samples, water separation index randomly changed from time period to time period, neither consistently increasing nor decreasing with time.

#### 6. Lubricity

The fuels submitted for the Ball-On-Cylinder Lubricity Evaluator (B.O.C.L.E.) were Series I fuels containing maximum allowable and minimum effective concentrations of corrosion inhibitor. Additionally, the control fuels for Series I and II and the petroleum JP-4 (sample 1064) were tested. Each fuel sample was evaluated at five intervals: 0, 3, 6, 9, and 15 months. Originally the 6-month interval was not included, but was added subsequent to the excessive wear exhibited in the main fuel pump during endurance testing of the F100 engine with shale derived JP-4 (Reference 4). Presently, there is no specification requirement for fuel lubricity.

The Ball-On-Cylinder Lubricity Evaluator test consists of a loaded, stationary ball contacting a rotating cylinder. The ball is placed perpendicular to the shaft supporting the cylinder. The cylinder rotates in a rectangular reservoir of fuel establishing a boundary layer of fuel on the cylinder. It is this boundary film which provides lubrication between the ball and the cylinder. As a guide for fuel lubricating quality and based upon past experience with hardware test-

ing, the following guidelines have been followed:

<u>WSD, mm</u>	<u>Fuel Lubricating Quality</u>
0.00 to 0.35	Good
0.36 to 0.45	Marginal
> 0.45	Poor

The fuels which display the best lubricity were those that, in a consistent manner, provided WSDs of 0.00 to 0.35mm. Four corrosion inhibitors/lubricity improvers, identified as CI1 through CI4, were evaluated. Two fuels containing CI1 in the maximum allowable concentrations exhibited good lubricity. Two similar fuels with maximum CI1 levels (0711 and 0712) were marginal at only the zero month.

There were fuels which displayed poor lubricity, i.e., greater than .45mm WSD for all time intervals; none of these fuels contained corrosion inhibitor.

Those fuels which had average WSDs consistently in the 0.36 to 0.45mm range (at any point during the testing) were considered of marginal lubricity. The lubricating quality of these fuels is likely to be unsatisfactory for use of in lubricity-sensitive systems such as the TF-30 or F100 engines.

Results for Series I control fuels, containing no corrosion inhibitor, varied widely (Fig. 3) showing B.O.C.L.E. ability to indicate poor lubricity, but not with the accuracy experienced when corrosion inhibitors are present.

For fuels with minimum corrosion inhibitors (Fig. 4), CI2 and CI1 performed consistently better than CI4 and CI3. Neglecting some early test program inconsistencies, the maximum allowable concentrations of

all the additives in Shale JP-4 displayed good lubricating characteristics by the 15-month test interval (Fig. 5).

There was no indication that any of the other additives, namely antioxidant, static dissipator additive or fuel system icing inhibitor, were effective as a lubricity enhancer.

The corrosion inhibitor which displayed the most effectiveness over the 15-month test was CI1 (Fig 6). With the minimum effective concentration, only the 0-month and the 3-month interval test showed marginal lubricity behavior. At the end of the test period, both minimum effective and maximum allowable concentrations exhibited good lubricity.

CI3 exhibited more erratic behavior (Fig 7). The differences exhibited at the 3-month interval were probably due to inherent repeatability problems in generating the wear scar diameters. The fuel sample (1146) which contained the minimum effective concentration did fall within the marginal area after completion of 15 months in storage. The CI4 sample displayed similar results (Fig 8).

The CI2 sample performed well at the maximum allowable level (Fig 9). The minimum effective concentration of CI2 exhibited minimally acceptable behavior. At the 3-month test interval, it surpassed the marginal level of lubricity. By the fifteenth month, though, it had a marginally acceptable WSD.

At the end of the 15-month interval, all fuels with the minimum effective concentration of corrosion inhibitor displayed marginally acceptable lubricity and could be used appropriately in those systems which are not considered lubricity sensitive. However, the optimum concentration was not determined for each of the corrosion inhibitors.

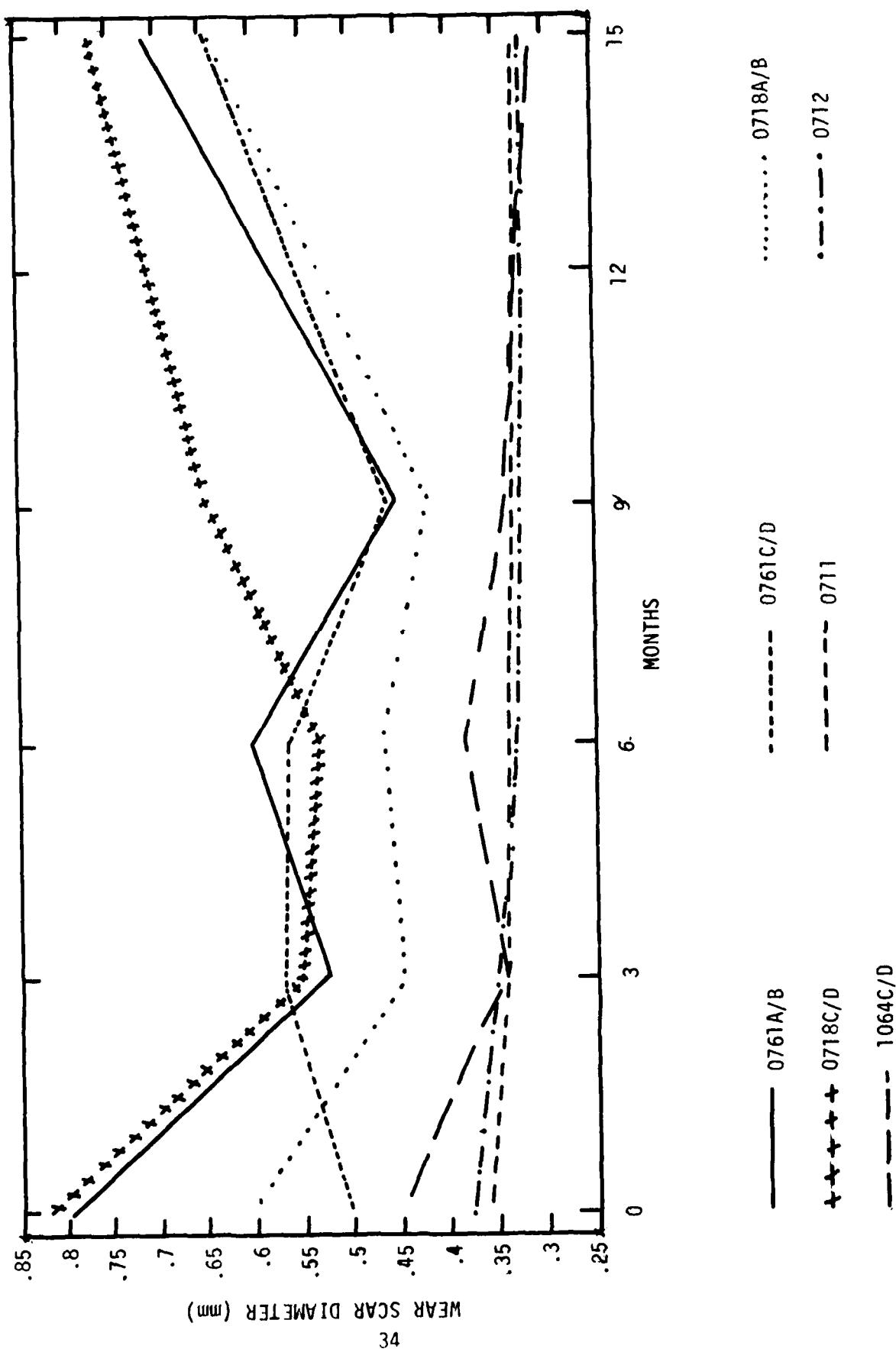


Figure 3. Series I Control Fuels B.O.C.L.E. Results

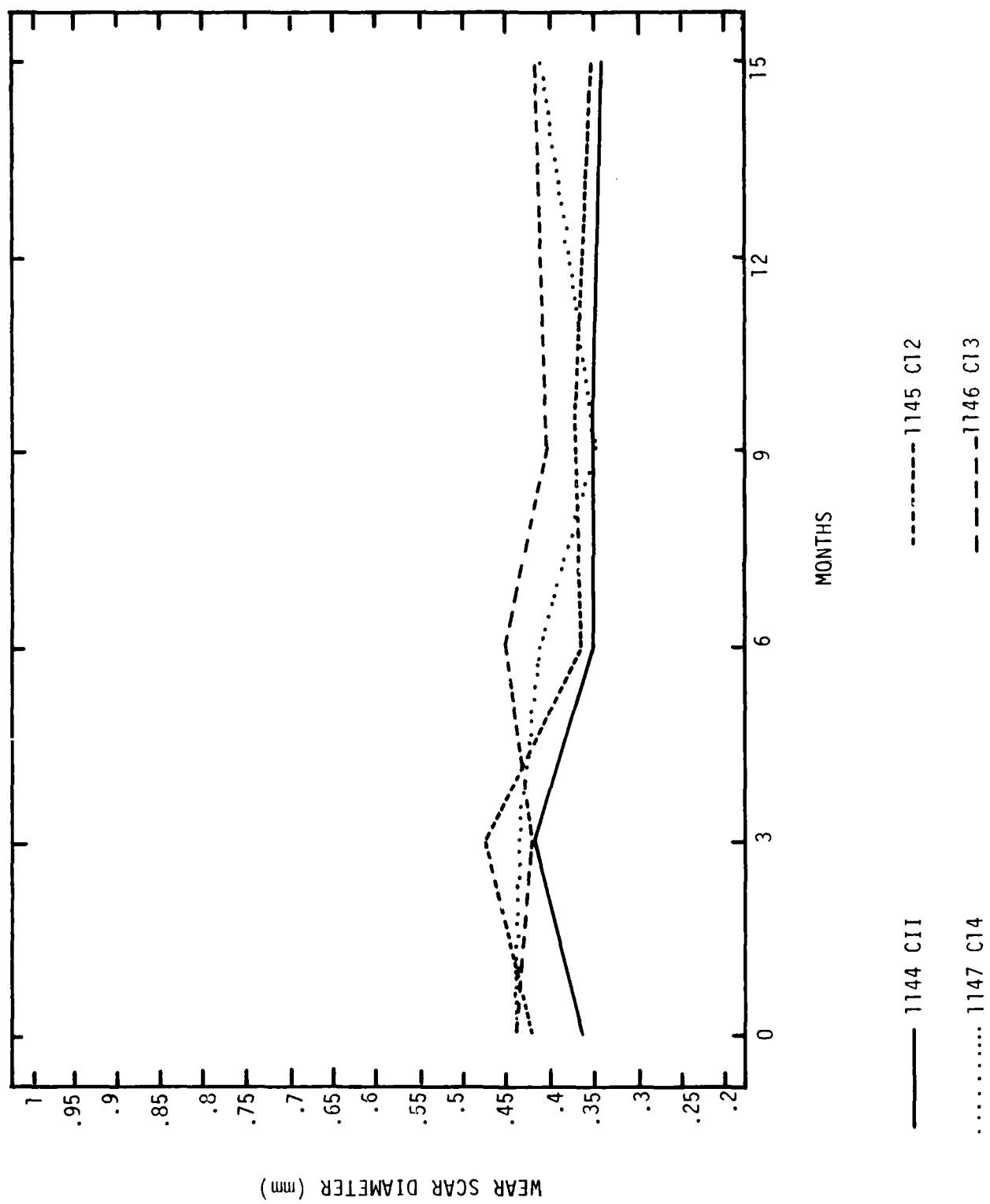


Figure 4. Minimum Corrosion Inhibitor B.O.C.L.E. Results

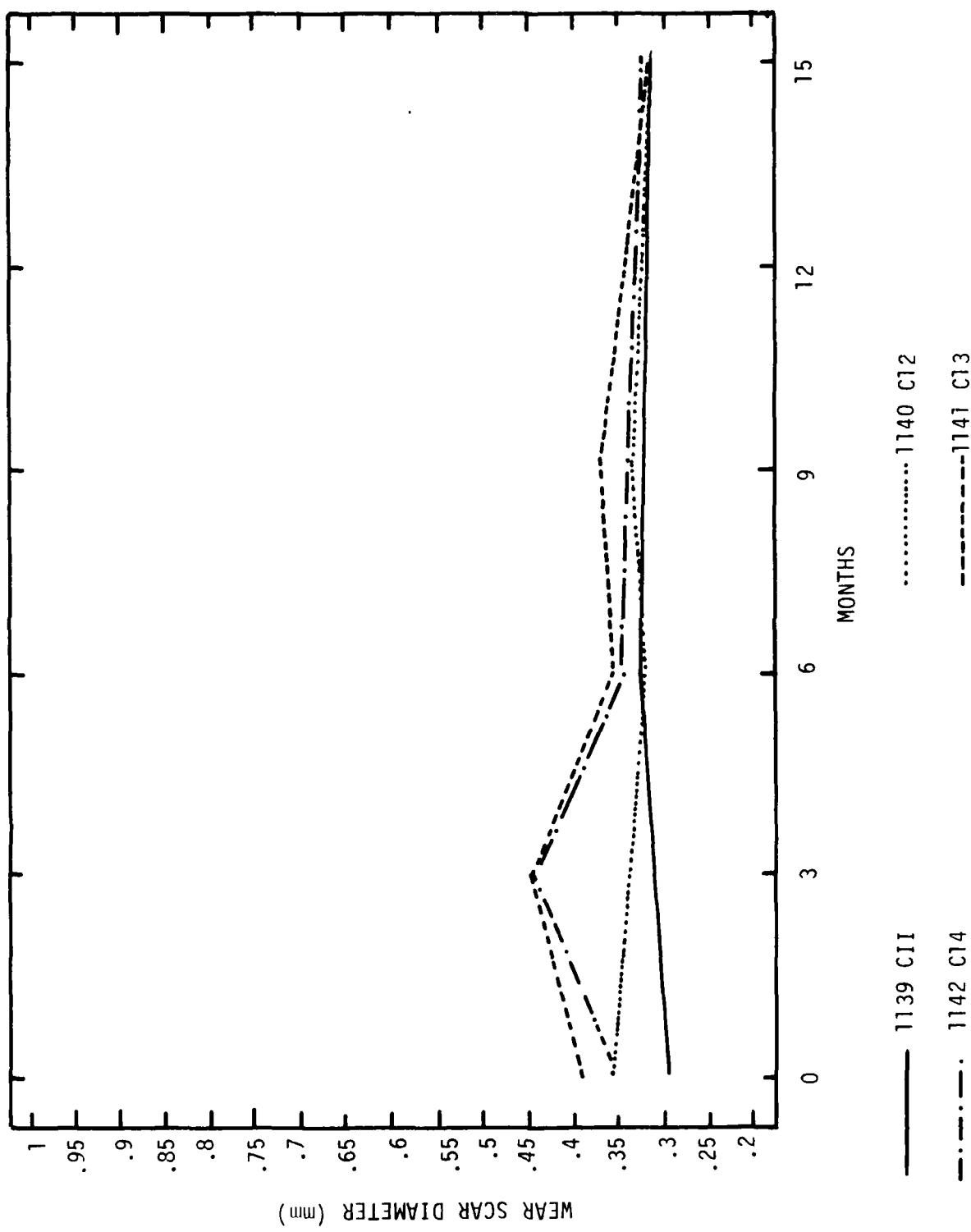


Figure 5. Maximum Corrosion Inhibitor B.O.C.L.E. Results

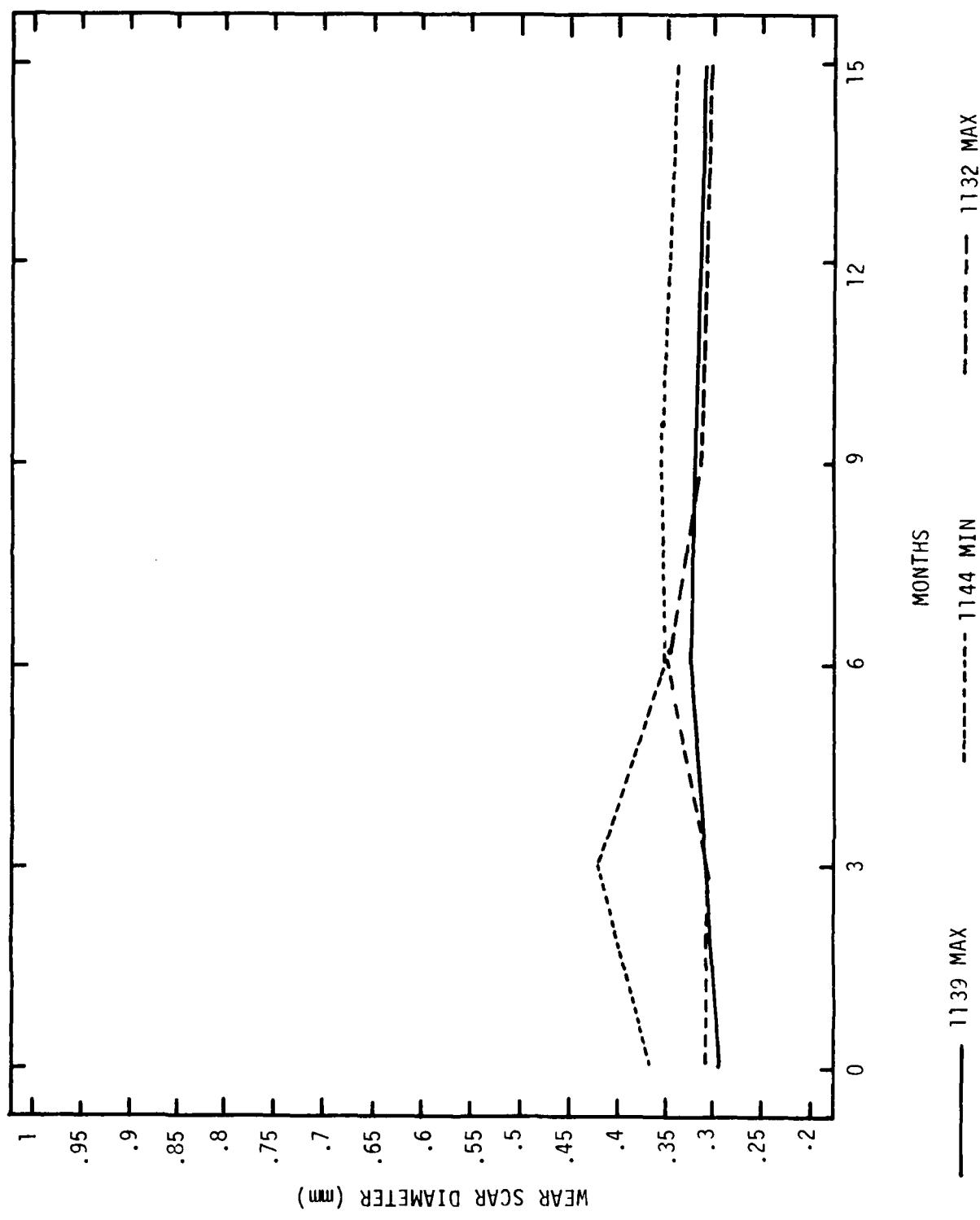


Figure 6. CII B.O.C.L.E. Results

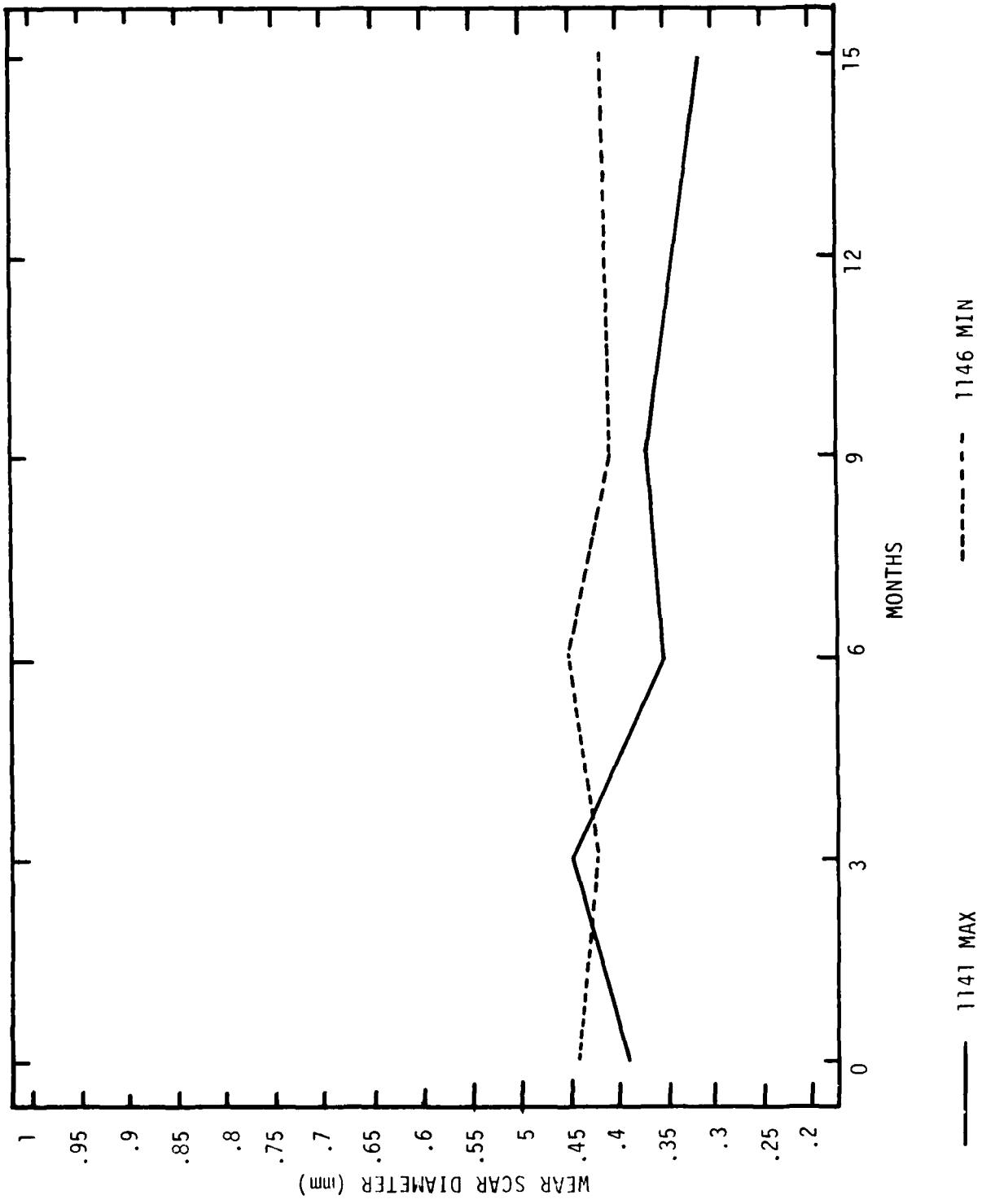
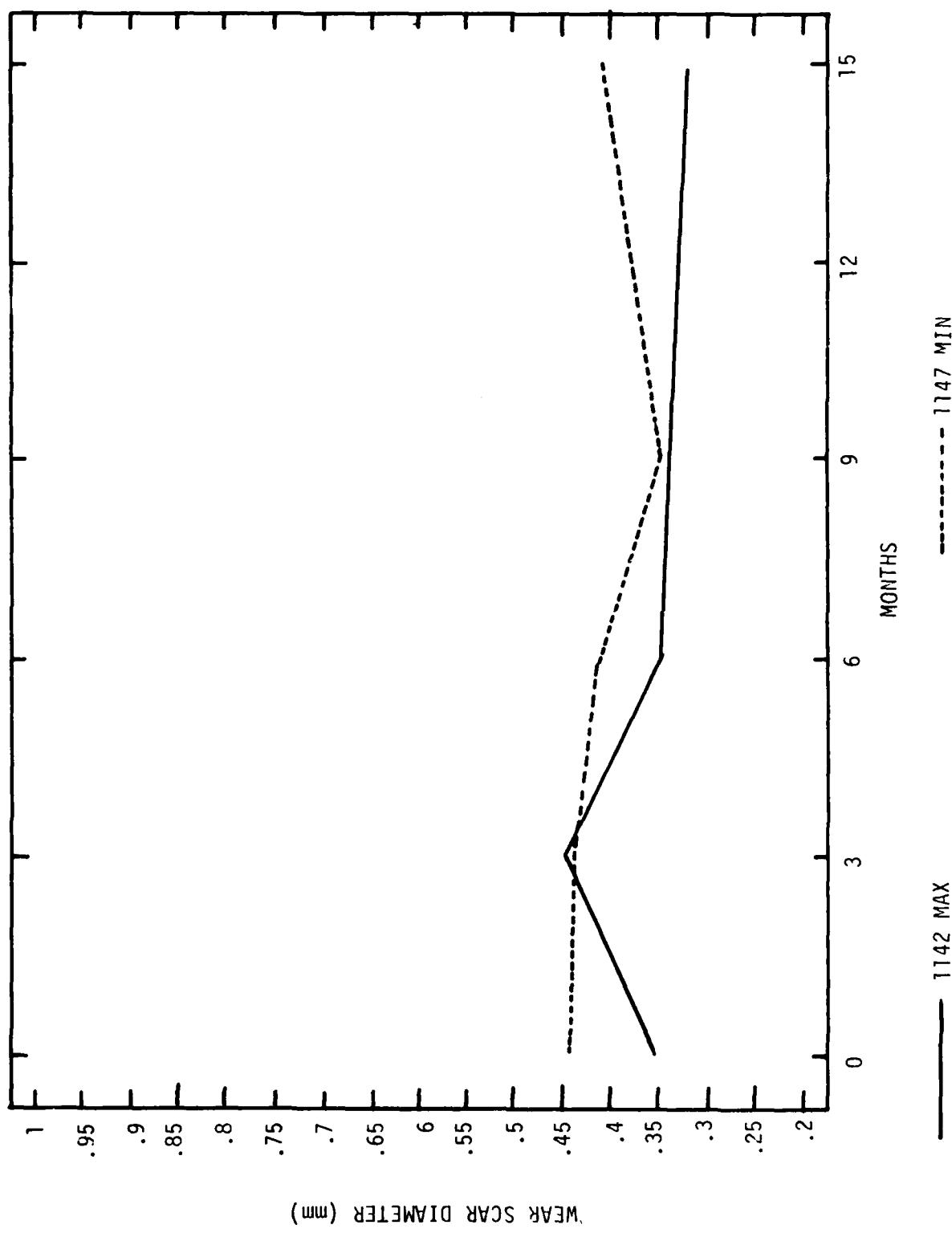


Figure 7. C13 B.O.C.L.E. Results

Figure 8. C14 B.O.C.L.E. Results



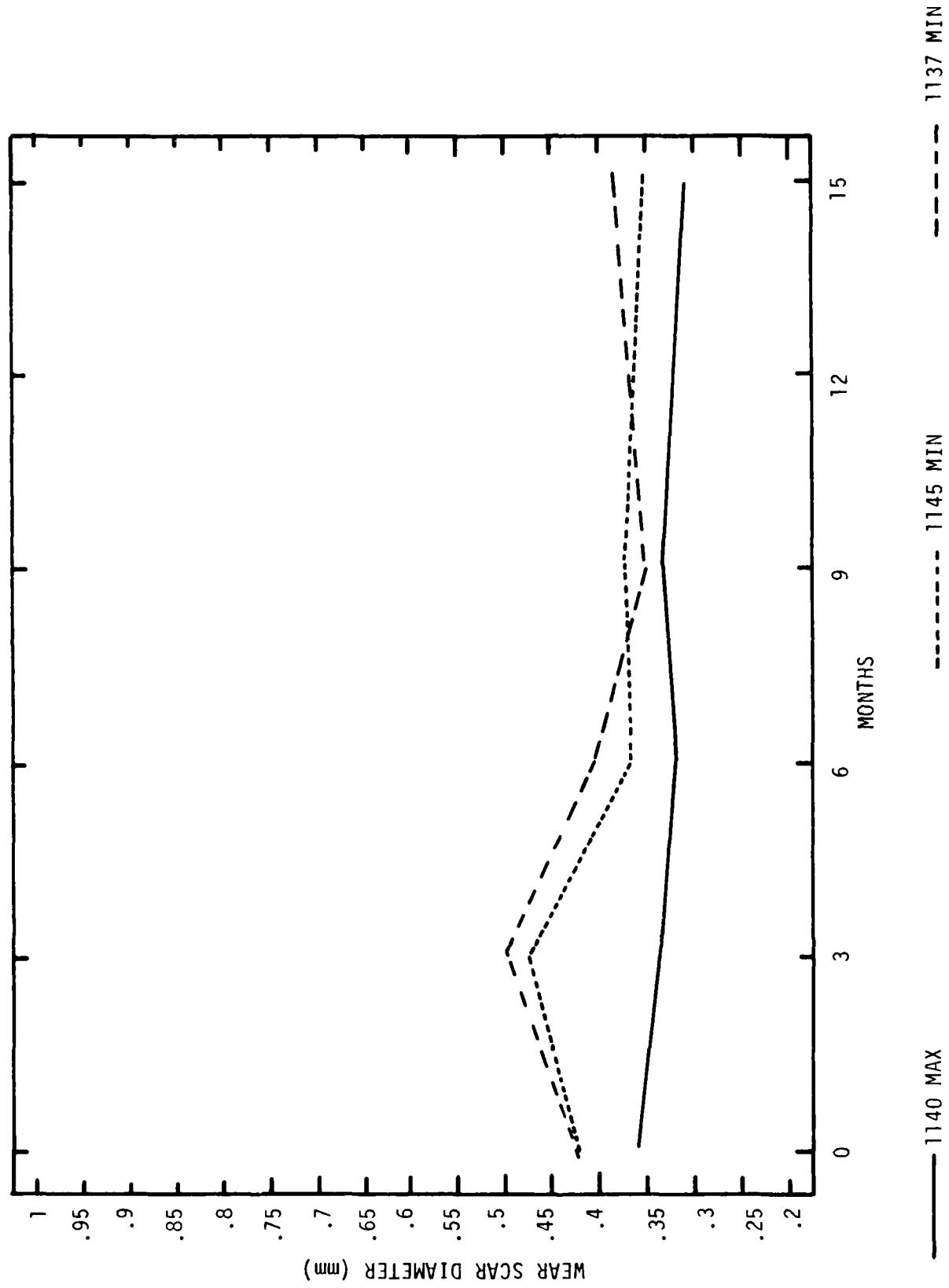


Figure 9. C12 B.O.C.L.E. Results

## 7. Electrical Conductivity

Fuel electrical conductivity was measured using a portable hand-held meter as prescribed by ASTM D2624, "Electrical Conductivity of Aviation Fuels Containing a Static Dissipater Additive." The JP-4 specification requires 200 to 600 picosiemens per meter (pS/m) or Conductivity Units (CU). The electrical conductivity of the test fuel was initially measured for the original drums of fuel. This measurement was taken at WPAFB after the drums were prepared as described in the "Origin of Test Fuel" section of this report. The drums were stored in cold storage for 15 months, with the exception of 0711 and 0712. At 15 months, a final reading was taken for all of the drums. The conductivity of the Series I and Series II test samples made from these drums was measured at the ninth and fifteenth month.

By the end of the test program, six drums of the 19 tested by the Fuels Branch had an acceptable conductivity. Of the Series I and Series II test samples, only three, 0711 (a drum), 1133 and 0761 A/B had a conductivity greater than 200 CU. By SFTLA results, 0712 also had an acceptable conductivity. The failure of the fuel to meet conductivity requirements was probably due to non-pretreating of fuel cans and transfer of the fuel from drum to storage can to sample container. For example, at 15 months, drummed 0722 fuel had an average conductivity of 149 CU, while the more transferred five gallon cans of 0722 (A through D) had an average conductivity of 26 CU. After studying the results for all drums and test samples, no trend was found as to time, amount of additive, type of additive, or combination of additives.

## 8. Naphthalenes

Drummed fuel samples 0711, 0712, 0718, 0722 and 0761 were analyzed at the beginning of the test program for hydrocarbon type by Monsanto Research Corporation (Reference 5). A modified ASTM Method D2789 and Monsanto Method 21-PQ-83-63 were used. No naphthalenes were found in any of the five samples by either method.

## 9. Specification Samples

Two drums of shale fuel, samples 0711 and 0712, contained additives as required and allowed by the specification, including the maximum allowable amount of A02 and C11. These duplicates were stored for 15 months, one in cold storage (0711) and one in outdoor storage (0712). Samples from these drums were tested at each of the test periods to determine if they met MIL-T-5624L specifications for JP-4 (Table 9).

Both fuels performed well and met the specification with few exceptions. In the ninth month, 0711 failed to meet requirements for Water Separation Index, as did 0712 in the zero and 15th month. These unsatisfactory results may be attributed to the additives, since both contained anti-static additive and maximum allowable corrosion inhibitor. These fuels received high water separation ratings at three months. A duplicate five gallon sample (1139) passed with more than marginal ratings. Sample 0711 failed to meet conductivity requirements at the three month test. This was probably due to instrument inaccuracies or temperature differences, since the fuel met specification for all other time periods.

TABLE 9. SPECIFICATION FUEL TEST RESULTS

	M O N T H S							
	0	3	9	15	0711	0712	0711	0712
Color (Saybold)	+30	+30	+30	+30	+30	+30	+30	+30
Total Acid No., mg KOH/g	0.004	0.005	0.008	0.008	0.005	0.006	0.005	0.006
Aromatics, vol %	12.1	12.0	11.0	10.9	11.9	11.5	12.1	12.1
Olefins, vol %	0.9	0.7	0.8	0.7	0.7	0.5	1.2	0.9
Mercaptan Sulfur, wt %	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Sulfur, Total, wt %	0.00	0.00	0.01	0.02	0.01	0.00	0.00	0.00
Distillation IBP deg C	24	24	24	24	24	24	24	24
10% Rec deg C	69	69	68	68	69	69	68	68
20% Rec deg C	101	101	100	100	101	101	100	100
50% Rec deg C	158	157	155	155	155	155	156	156
90% Rec deg C	219	219	217	217	217	217	218	218
Final Boiling Pt deg C	249	249	247	248	247	248	253	251
Density, kg/Liter	0.765	0.765						
Gravity, API			53.6	53.5	53.6	53.6	53.5	53.5
Vapor Pressure, kPa (psi)	19		(2.9)	(2.9)	(2.8)		(3.0)	(3.0)
Freezing Point, deg C (deg F)	-73	-70	(B-99)	(B-99)	(B-99)	(-95)	(B-99)	(-96)
Net Heat of Combustion, MJ/kg	43.5	43.5						
Hydrogen Content, wt %	14.3	14.3						
Smoke Point, mm					27	27		
Copper Strip Corrosion	1A	1A	1A	1A	1A	1A	1A	1A
Thermal Stability at 260°C								
Change in Pressure Drop, mm of Hg	0	0	0	0	0	0	0	0
Preheater Deposit Code	1	1	1	1	1	1	1	1
TDR Rating Code	1	1	1	1	0	0	0	1
Existent Gum, mg/100ml	0.4	0.0	2.0	0.4	0.2	1.6	0.0	1.0
Particulate Matter, mg/L	0.1	0.1	0.0	0.0	0.2	0.0	0.2	0.3
Filtration Time, minutes	5	5	6	6	5	5	5	5
Water Reaction Interface	1	1	1	1	1	1	1	1
Minisonic	74	66	83	85	69	70	77	66
Fuel System Icing Inhib, vol%	0.13	0.12	0.12	0.10	0.13	0.13	0.13	0.13
Conductivity, pS/m(CU)	200	295	180	200	290	300	265	260

## V. CONCLUSIONS

The shale JP-4 fuel in this test program performed very well in the areas of thermal and storage stability. Fuel lubricating quality was unacceptable without corrosion inhibitor. While some antioxidant and corrosion inhibitor additives performed better than others, no additives outside those listed in the JP-4 specification were required.

Specification tests that were performed throughout the program were JFTOT, particulates/filtration time, and existent gum. All fuel samples met the specified limits for these tests for all test times. All test samples were tested for electrical conductivity at nine and fifteen months and most were low, except two samples that were in their original container throughout the test. These had acceptable conductivity, suggesting that the fuel itself would perform well. In a minisonic test, used to evaluate water separation characteristics, all samples had an acceptable water separation at most time periods, except those containing JFA-5. Corrosion inhibitor caused some results to be intermittently below requirements.

In non-specification tests, the peroxide level was low for all samples and non-existent for some, except those not containing antioxidant. For lubricity evaluation using the Ball-on-Cylinder Lubricity Evaluator, the shale fuel did not perform well unless a maximum concentration of corrosion inhibitor was present.

The best performing antioxidants overall were A02, A06 and A05. This is considering the adverse effect of A01 with maximum corrosion inhibitor on JFTOT results and the peroxides found in samples containing

A07, A08 and A09 in either the minimum or maximum concentrations. A09, the resorcinol antioxidant, also adversely affected water separation at two times the maximum concentration. A04 had JFTOT results bordering on unacceptable until the fifteenth month, and at the fifteenth month, peroxides were beginning to form. Peroxide test results showed that the tertiary butyl phenolic antioxidants were the most effective in preventing peroxidation in the shale fuel. This correlation did not extend to JFTOT results.

Considering the four corrosion inhibitor/lubricity additives, CI1 performed the best in Ball-on-Cylinder tests at the maximum concentration. It did not do as well in samples in outdoor or cold storage as it did in samples stored at room temperature. CI3 at maximum concentration adversely affected JFTOT results and maximum CI3 with A01 antioxidant showed some existent gum. Maximum CI1 and CI2 performed better in the lubricity evaluation than maximum CI4 and CI3, and similarly for the minimum concentrations.

The JFA-5 and NDA additives improved thermal stability but were not required for the test fuels to meet JP-4 specification requirements for the thermal stability. JFA-5 adversely affected water separation.

Concluding, then, shale JP-4 fuel was, for the five year simulated test period, a quality jet fuel. To maintain high quality, a minimum amount of antioxidant is required to prevent peroxidation and a maximum concentration of corrosion inhibitor is required to improve lubricity. Conductivity of the fuel should be monitored.

## REFERENCES

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2. D.S. Duvall, "Analysis of a Deposit Found in a Shale JP-4 Sample," Technical Operating Report No. 2035-072, AF Contract Number: F33615-81-C-2035, Monsanto Research Corporation, 15 December 1983.
3. W.G. Dukek, "Test Programs and Methods for Water Separation Characteristics of Aviation Fuels," ASTM Research Report, File No. RR D-2-1050; Sponsoring Committee D-2 TD J-X, 1 September 1983.
4. Weston, J.L., Part III, Accelerated Mission Test Using Shale Oil Derived JP-4 Part II-F100 Aviation Gas Turbine, AFWAL-TR-84-2092, United Technologies Corporation, Pratt & Whitney Engineering Division, Florida, September 1984.
5. D.S. Duvall and A.D. Snyder, "Determination of Naphthalenes in Shale Fuels," Technical Operating Report No. 2035-076, AF Contract Number F33615-81-C-2035, 23 December 1983.

APPENDIX A DRUM TO TEST SAMPLE RELATIONSHIP

SHALE ADDITIVE PROGRAM

DRUM POSF	SAMPLE ORIGIN DAUGHTER CAN POSF
0714	1150, 1131
0716	1152, 1155, 1154, 1155
0717	1156, 1157, 1158
0718	1159, 1140, 0718 (2 CONTROLS)
0719	1141, 1142, 1143, 1144
0720	1145, 1146, 1147, 1148
0721	NONE
0722	0722 (2 CONTROLS)
0723	0723, 1149
0724	0724, 1150
0725	0725, 1151
0726	0726, 1152
0727	0727, 1155
0728	0728, 1154
0729	0729, 1155
0730	0730, 1156
0761	0761 (2 CONTROLS)
0762 - 0768	COLD STORAGE

**APPENDIX B**  
**TEST RESULTS BY TEST**

TABLE B-1. SERIES I (ROOM TEMPERATURE STORAGE) JFTOT TEST RESULTS

FUEL CODE	TEMP (°C)	MONTHS											
		0		3		9		15					
		VISUAL CODE	Δ TDR	ΔΦ, mmHg	VISUAL CODE	Δ TDR	ΔP, mmHg	VISUAL CODE	Δ TDR	ΔΦ, mmHg	VISUAL CODE	Δ TDR	ΔΦ, mmHg
0711	310	B.P.	B.P.	B.P.	1	11	2	1	5	*	B.P.	B.P.	B.P.
0712	310	B.P.	B.P.	B.P.	2	17.5	0	1	2	0	B.P.	B.P.	B.P.
0718 A/B	310	2	9.3	0	2	13	0	1	3	0	1	1	0
0718 C/D	310	1	2.5	0	2	7	0.3	1	2	0	1	17.5	0
0761 A/B	310	1	5	0	2	9.5	0	2	10	0	1	2	0
0761 C/D	310	1	2	0.1	1	7	0	1	4	0	1	4	0
1064 C/D	310	1	2.5	2	1	4	0	1	4	0.2	2	11	0.2
1130 A/B	310	2	5	0.5	4	13	0.3	1	5.5	0.1	3	11.5	0.1
	290	-	-	-	1	3.4	0.1	-	-	-	1	1.5	0.2
1131 A/B	310	2+	12	0	3+	20.5	5	1	7	0	2	8	0
	290	-	-	-	1	1.5	0	-	-	-	-	-	-
1132 A/B	310	2	8.5	0	4	10	0	2	15	0	4+	9	0
	290	-	-	-	2	2	0	-	-	-	1	4	0
1133 A/B	310	4	7.5	0	4	23.5	0	1	2	0	2	4	0
	290	1	1	0	1	1	0	-	-	-	-	-	-
1134 A/B	310	1	2.5	0	3	5	0.1	2	5	*	1	1.5	0
	290	-	-	-	1	4	0.1	-	-	-	-	-	-
1135 A/B	310	2	5.5	1	1	5	0	1	7	0	1	3	0
1136 A/B	310	1	4.2	0	1	1	0	1	3	0	1	4.5	0

TABLE B-1. SERIES I (ROOM TEMPERATURE STORAGE) JFTOT TEST RESULTS (CON'T)

MONTHS

FUEL CODE	TEMP (°C)	0			3			9			15		
		VISUAL CODE	ΔTDR ΔP, mmHg	VISUAL ΔTDR CODE	VISUAL ΔTDR ΔP, mmHg	VISUAL CODE	ΔTDR ΔP, mmHg						
1137 A/B	310	1	3.8	0	1	1.5	0	1	4.5	*	1	6	0.3
1138 A/B	310	1	4	0.3	3	3	0.2	1	1.5	*	1	2	0
	290	-	-	-	1	1.5	0	-	-	-	-	-	-
1139 A/B	310	2	14	0.3	1	10	0.1	1	0	0.3	2	10	0
1140 A/B	310	2	10.5	0.2	1	11	0	2	11.5	0.4	1	4.5	0
1141 A/B	310	2	2.5	0	1	13	0.2	1	11.5	*	3	14	0.2
	290	-	-	-	-	-	-	-	-	-	1	2	0
1142 A/B	310	1	3	0.2	2	10.5	0	1	3	*	2	11	0.1
1143 A/B	310	1	3.5	0.2	1	2.5	0	1	2	0	1	4	0
1144 A/B	310	1	5.6	0	2	10.5	0	2	14	*	2	4.5	0
1145 A/B	310	1	10.5	0	1	5	0	1	9	0.2	1	5	0.1
1146 A/B	310	1	4	0.4	2	18	0.1	1	1	0.2	2	9	2
1147 A/B	310	1	4.8	0.4	2	12	0.1	1	4	0.3	1	7	0
1148 A/B	310	2+	4.8	0	1	7	0	1	6	*	1	4	0

B.P. = Break Point Test

\* = Pressure Transducer Non-Operative

TABLE B-2. SERIES II (110°F STORAGE) JFTOT TEST RESULTS

FUEL CODE	TEMP (°C)	MONTHS						VISUAL CODE	ΔTDR ΔP, mmHg	VISUAL ΔTDR ΔP, mmHg	VISUAL CODE	ΔTDR ΔP, mmHg	VISUAL CODE	ΔTDR ΔP, mmHg
		0	3	9	15									
0722 A/B	320	1	8.5	0	2	19.5	0	2	4.5	0	4+	19	0.9	
	300	-	-	-	-	-	-	-	-	-	3+	19	0.3	
	280	-	-	-	-	-	-	-	-	-	1	3.5	0	
0722 C/D	320	2	10	3	2	10.5	0	1	4	0	4+	29	0	
	300	-	-	-	-	-	-	-	-	-	4	15	C	
	280	-	-	-	-	-	-	-	-	-	1	7	0	
0723 A/B	320	2	24.2	0.3	2	20	0.1	1	3	*	1	4.5	0	
0724 A/B	320	4	26.6	0	2	2	0	3	24	0	1	3.5	0.1	
	300	1	1.5	0	-	-	-	1	3	0	-	-	-	
0725 A/B	320	2+	20	2	1	12	0	2	11	0	1	2	0	
0726 A/B	320	2	8.3	0.2	3	21	0.3	1	5	0.2	1	4	0	
	300	-	-	-	1	2.5	0	-	-	0	-	-	-	
0727 A/B	320	4	27.5	0.4	2	16	0	1	9	0.2	2	9	0.1	
	300	1	2.5	0	-	-	-	-	-	-	-	-	-	
0728 A/B	320	4	24.5	1	4+	30	0	2	12	0	2	14	0	
	300	1	6.3	0	1	8	0	-	-	-	-	-	-	
0729 A/B	320	1	3.5	0.2	2	18	0	1	1	0.2	2	6.3	0.2	
0730 A/B	320	4	15.8	0.1	4+	31	0	3	12	0	4	39.5	0	
	300	1	1.5	0.3	1	2	0	1	2	0	1	2	0.2	
1064 A/B	320	3	14	0	4	22	0	1	11	0	1	3	1	
	300	1	6.5	1	-	3	0	-	-	-	-	-	-	
1149 A/B	320	4+	33	0	2	15	1	1	9.5	0	2	6	0	
	300	2	9.5	0.2	-	-	-	-	-	-	-	-	-	

TABLE B-2. SERIES II (110°F STORAGE) JFTOT TEST RESULTS (CONT)

FUEL CODE	TEMP (°C)	MONTHS											
		0		3		9		15					
		VISUAL CODE	ΔTDR ΔP, mmHg		VISUAL CODE	ΔTDR ΔP, mmHg		VISUAL CODE	ΔTDR ΔP, mmHg		VISUAL CODE	ΔTDR ΔP, mmHg	
1150 A/B	320	2	3.9	1	4+	31	0.1	1	5	0	3	17	0
	300	-	-	0	1	3	0.2	-	-	-	1	2.5	0.2
1151 A/B	320	2+	13	0	3	27.5	0	1	9	0	1	5	0
	300	-	-	-	1	0.5	1.5	-	-	-	-	-	-
1152 A/B	320	3	14	0	2	16	0	2	7	0	1	4	0
	310	2	12.5	0	-	-	-	-	-	-	-	-	-
1153 A/B	320	2	10.5	0.2	2	20	0.9	1	1	0	2	4	0.3
	310	-	-	-	4+	22.5	0.1	2	15.5	0	2	7	0
1154 A/B	320	4	25	0.6	-	-	-	-	-	-	-	-	-
	310	1	5	0.1	1	1.5	0	-	-	-	-	-	-
1155 A/B	320	2	7.5	0.1	2	6.5	0	1	0.5	0.4	4	21	0
	300	-	-	-	-	-	-	-	-	-	1	1.8	0
1156 A/B	320	4+	20	0.3	3	10.5	0	4	19	0.1	4	11	0.1
	300	1	2	0	1	0.5	0.1	1	2	0	1	3.5	0.2
1710 A/B	320	-	-	-	1	3	0.4	1	7	0	1	1	0
1711 A/B	320	-	-	-	1	7.5	1	1	3	0	2	10	0

TABLE B-3. PARTICULATE/FILTRATION TIME TEST RESULTS

FUEL CODE	PARTICULATES(mg/l)/FILTRATION TIME(min)			
-----------	---	--	--	--

MONTHS				
	0	3	9	15
0711	0.1/5	0.0/6	0.2/5	0.2/5
0712	0.1/5	0.0/6	0.0/5	0.3/5
0718 A/B	0.1/4	0.2/6	0.0/5	0.1/5
0718 C/D	0.1/4	0.1/6	0.1/5	0.1/5
0722 A/B	0.2/4	0.2/6	0.2/5	0.5/3
0722 C/D	0.1/4	0.2/6	0.2/5	1.0/5
0723 A/B	0.1/4	0.2/6	0.2/5	0.1/4
0724 A/B	0.1/4	0.2/6	0.3/5	0.2/5
0725 A/B	0.2/4	0.2/6	0.2/5	0.1/4
0726 A/B	0.2/4	0.2/6	0.1/5	0.1/4
0727 A/B	0.1/4	0.2/6	0.2/6	0.2/5
0728 A/B	0.1/4	0.2/5	0.1/5	0.2/5
0729 A/B	0.1/4	0.1/5	0.2/5	0.2/5
0730 A/B	0.1/5	0.1/6	0.2/6	0.1/4
0761 A/B	0.1/5	0.1/6	0.2/5	0.1/4
0761 A/B	0.1/5	0.3/6	0.2/5	0.1/4
1064 A/B	0.2/5	0.2/5	0.2/5	0.1/4
1064 C/D	0.1/5	0.2/6	0.1/5	0.1/4
1130 A/B	0.2/5	0.1/5	0.3/8	0.1/5
1131 A/B	0.1/4	0.1/4	0.1/5	0.1/5
1132 A/B	0.2/4	0.2/6	0.4/6	0.2/5
1133 A/B	0.3/5	0.1/5	0.2/5	0.3/5
1134 A/B	0.2/5	0.1/5	0.1/6	0.1/4
1135 A/B	0.3/5	0.1/6	0.3/6	0.2/4
1136 A/B	0.2/4	0.1/5	0.3/6	0.2/4
1137 A/B	0.5/4	0.1/6	0.2/6	0.3/4
1138 A/B	0.1/3	0.1/7	0.2/6	0.2/5
1139 A/B	0.1/3	0.1/6	0.2/5	0.3/4
1140 A/B	0.4/5	0.2/6	0.1/5	0.1/4
1141 A/B	0.3/5	0.2/6	0.2/5	0.2/4
1142 A/B	0.2/4	0.1/6	0.0/5	0.2/4
1143 A/B	0.2/5	0.3/6	0.2/6	0.3/5
1144 A/B	0.3/5	0.3/6	0.1/6	0.2/4
1145 A/B	0.3/5	0.2/6	0.2/6	0.3/4
1146 A/B	0.2/4	0.1/6	0.1/5	0.1/5
1147 A/B	0.3/4	0.0/6	0.1/6	0.2/5
1148 A/B	0.1/4	0.2/7	0.1/6	0.3/5
1149 A/B	17.2/4	0.1/5	0.2/5	0.3/5
1150 A/B	0.1/4	0.2/5	0.3/5	0.2/5
1151 A/B	0.2/5	0.0/6	0.5/5	0.2/5
1152 A/B	0.1/4	0.0/6	0.3/6	0.2/5
1153 A/B	0.2/4	0.2/6	0.2/6	0.2/5
1154 A/B	0.2/4	0.1/4	0.4/5	0.2/4
1155 A/B	0.2/4	0.1/6	0.1/6	0.2/4
1156 A/B	0.2/4	0.1/5	0.3/5	0.2/4
1710 A/B	---	0.2/5	0.2/6	0.1/4
1711 A/B	---	0.4/4	0.3/5	0.3/5

TABLE B-4. EXISTENT GUM TEST RESULTS

FUEL CODE	EXISTENT GUM (mg/100ml)			
	MONTHS			
	0	3	9	15
0711	0.4	2.0	0.2	0.0
0712	0.0	0.4	1.6	1.0
0718 A/B	0.2	0.0	0.4	0.0(A)
0718 C/D	0.0	0.0	0.4	0.6
0722 A/B	0.2	0.0	0.4	0.0
0722 C/D	0.0	0.6	0.0	3.4
0723 A/B	0.4	0.6	0.6	0.2
0724 A/B	0.0	0.4	0.0	0.6
0725 A/B	0.0	0.4	1.2	0.0
0726 A/B	0.0	0.2	0.6	0.6
0727 A/B	0.6	0.0	0.6	0.2
0728 A/B	0.0	0.8	0.2	0.4
0729 A/B	0.6	0.2	0.0	0.4
0730 A/B	0.6	0.4	0.4	0.2
0761 A/B	0.6	0.4	0.6	0.6
0761 C/D	0.4	0.0	1.0	0.0
1064 A/B	0.2	0.0	0.0	0.2
1064 C/D	0.0	0.0	0.0	0.0
1130 A/B	0.6	0.0	1.0	0.6
1131 A/B	0.0	0.4	0.0	0.4
1132 A/B	0.4	0.4	0.0	0.0
1133 A/B	0.0	0.6	0.0	0.2
1134 A/B	0.0	0.2	0.2	3.2
1135 A/B	0.6	0.0	0.0	0.0
1136 A/B	0.6	0.4	1.2	0.6
1137 A/B	0.4	0.0	0.2	0.4
1138 A/B	0.8	0.6	0.6	0.6
1139 A/B	0.8	0.0	0.6	0.0
1140 A/B	0.0	0.0	0.0	0.0
1141 A/B	0.6	0.4	0.0	0.0
1142 A/B	0.6	0.8	0.8	0.8
1143 A/B	0.0	0.0	0.0	0.2
1144 A/B	0.0	0.4	0.4	0.2
1145 A/B	0.0	0.4	0.4	0.2
1146 A/B	0.4	0.0	0.0	0.4
1147 A/B	0.4	0.2	0.4	0.0
1148 A/B	1.0	0.0	0.2	0.0
1149 A/B	0.0	0.2	0.4	0.0
1150 A/B	0.2	0.0	0.4	0.0
1151 A/B	0.0	0.2	0.6	0.0
1152 A/B	0.0	0.0	0.8	0.0
1153 A/B	0.0	0.0	0.0	0.0
1154 A/B	0.4	0.8	0.2	0.2
1155 A/B	0.2	0.4	0.6	0.2
1156 A/B	0.0	0.8	0.4	0.0
1710 A/B	---	0.6	0.8	0.0
1711 A/B	---	0.4	0.6	0.8

TABLE B-5. PEROXIDE TEST RESULTS

<u>FUEL CODE</u>	<u>PEROXIDE NUMBER ( ppm )</u>			
	MONTHS			
	0	3	9	15
0711	0	0	0	0
0712	0	0	0	0
0718 A/B	0.140	0	0	0
0718 C/D	0.140	0	0.320	0
0722 A/B	0	0.302	0.640	1018.89 1073.69
0722 C/D	0	0.795	2.200 2.400	538.64 (C) 460.15 (C) 828.57 (D) 1004.77 (D)
0723 A/B	0	0	0	0
0724 A/B	0	0	0	0.415
0725 A/B	0	0	0	0.520
0726 A/B	0.279	0	0	0
0727 A/B	0	0	0.320	.526
0728 A/B	0	0	0.160	1.442 1.420
0729 A/B	0	0.087	0	5.560 4.420
0730 A/B	0	0	0	0
0761 A/B	0	0	0.080	0
0761 C/D	0.356	0	0	0
1064 A/B	0.106	0	0.080	1.151 0.720
1064 C/D	0.074	0	0.240	0
1149	0	0	0	0

TABLE B-5. PEROXIDE TEST RESULTS (Con't)

<u>FUEL CODE</u>	<u>PEROXIDE NUMBER ( ppm )</u>			
	<u>MONTHS</u>			
	0	3	9	15
1150 A/B	0	0	0	0
1151 A/B	0	0	0	0.525
1152 A/B	0	0	0	0
1153 A/B	0	.016	.960	0.701
1154 A/B	0	0	0.480	1.586 1.830
1155 A/B	0	0	3.400 3.000	6.592 5.710
1156 A/B	0.070	0.141	0.320	0.841
1710 A/B	-	0	0.640	1.553 1.740
1711 A/B	-	0	0.880	1.783 2.830

\* The minimum detectable limit for the ASTM Peroxide Determination procedure is not known at this time. A 0 ppm peroxide number indicates that there was no notable color change to the sample with the addition of the KI solution and starch making titration with the sodium thiosulfate impossible.

TABLE B-6. MINISONIC TEST RESULTS

FUEL CODE	WATER SEPARATION INDEX			
	MONTHS			
	0	3	9	15
0711	74	83	69	77
0712	66	85	70	66
9718 A/B	90	99	88	93
0718 C/D	90	99	93	97
0722 A/B	78	88	95	72
0722 C/D	90	85	94	56
0723 A/B	74	96	100	91
0724 A/B	79	96	96	93
0725 A/B	85	91	90	93
0726 A/B	74	88	92	84
0727 A/B	78	84	90	85
0728 A/B	77	99	93	72
0729 A/B	82	96	96	81
0730 A/B	63	93	89	93
0761 A/B	92	97	92	99
0761 C/D	89	99	89	90
1064 A/B	67	98	97	97
1064 C/D	85	79	86	89
1130 A/B	75	81	82	92
1131 A/B	88	86	88	95
1132 A/B	72	74	56	86
1133 A/B	81	90	65	73
1134 A/B	63	56	44	67
1135 A/B	71	61	58	67
1136 A/B	86	77	60	81
1137 A/B	91	95	79	88
1138 A/B	39	57	41	56
1139 A/B	87	88	67	95
1140 A/B	78	87	81	89
1141 A/B	73	81	63	76
1142 A/B	66	88	73	74
1143 A/B	85	91	57	67
1144 A/B	83	92	72	94
1145 A/B	84	93	77	90
1146 A/B	83	81	95	86
1147 A/B	90	90	77	88
1148 A/B	49	55	40	61
1149 A/B	80	94	87	85
1150 A/B	84	94	98	96
1151 A/B	92	96	90	98
1152 A/B	92	90	96	93
1153 A/B	82	75	86	95
1154 A/B	89	86	94	85
1155 A/B	82	93	92	75
1156 A/B	76	88	77	90
1710 A/B	--	88	81	89
1711 A/B	--	65	57	57

TABLE B-7. B.O.C.L.E. TEST RESULTS

<u>FUEL CODE</u>	<u>RUN #</u>	<u>WEAR SCAR DIAMETER (mm)</u>				
		<u>MONTHS</u>				
		0	3	6	9	15
0711	1	0.350	0.335	0.335	0.340	0.335
	2	0.400	0.350	0.330	0.330	0.330
	AVG	0.375	0.342	0.342	0.335	0.3325
0712	1	0.380	0.340	0.330	0.330	0.325
	2	0.375	0.365	0.335	0.325	0.325
	AVG	0.377	0.352	0.332	0.327	0.325
0718 A/B	1	0.575	0.430	0.470	0.460	0.750
	2	0.635	0.470	0.470	0.390	0.520
	3	-	-	-	-	-
	AVG	0.605	0.45	0.47	0.425	0.6483
0718 C/D	1	0.815	0.555	0.525	0.605	0.910
	2	0.805	0.550	0.545	0.700	0.560
	3	-	-	-	-	0.855
	AVG	0.810	0.522	0.535	0.6525	0.775
0722 A/B	1	0.405	0.435	0.320	0.380	0.375
	2	0.360	0.440	0.335	0.345	0.380
	AVG	0.382	0.437	0.327	0.362	0.377
	0722 C/D	1	0.490	0.440	0.310	0.340
0761 A/B	2	0.340	0.415	0.335	0.350	0.405
	AVG	0.410	0.427	0.322	0.345	0.410
	1	0.815	0.530	0.565	0.460	1.105
	2	0.760	0.520	0.650	0.445	0.605
0761 C/D	3	-	-	-	-	0.435
	AVG	0.787	0.525	0.607	0.452	0.715
	1	0.525	0.555	0.590	0.440	0.910
	2	0.480	0.590	0.540	0.490	0.615
T064 C/D	3	-	-	-	-	0.435
	AVG	0.502	0.572	0.565	0.465	0.653
	1	0.470	0.345	0.410	0.355	0.320
	2	0.435	0.345	0.365	0.335	0.315
T132 A/B	AVG	0.452	0.345	0.387	0.345	0.317
	1	0.310	0.310	0.345	0.320	0.300
	2	0.305	0.305	0.350	0.310	0.310
T137 A/B	AVG	0.307	0.307	0.347	0.315	0.305
	1	0.380	0.430	0.460	0.365	0.395
	2	0.470	0.560	0.375	0.365	0.365
T139 A/B	AVG	0.425	0.495	0.417	0.365	0.380
	1	0.270	0.310	0.345	0.325	0.320
	2	0.320	0.305	0.305	0.310	0.305
	AVG	0.295	0.307	0.325	0.317	0.312

TABLE B-7. B.O.C.L.E. TEST RESULTS (CON'T)

<u>FUEL CODE</u>	<u>RUN #</u>	<u>WEAR SCAR DIAMETER (mm)</u>				
		<u>MONTHS</u>				
		0	3	6	9	15
1140 A/B	1	0.370	0.335	0.330	0.335	0.315
	2	0.350	0.340	0.310	0.335	0.310
	AVG	0.360	0.337	0.320	0.335	0.312
1141 A/B	1	0.375	0.440	0.340	0.345	0.335
	2	0.405	0.455	0.370	0.395	0.265
	3	-	-	-	-	0.345
1142 A/B	AVG	0.390	0.447	0.355	0.370	0.315
	1	0.370	0.440	0.360	0.345	0.325
	2	0.335	0.450	0.335	0.335	0.320
1144 A/B	AVG	0.352	0.445	0.347	0.340	0.322
	1	0.365	0.430	0.345	0.350	0.330
	2	0.365	0.410	0.360	0.360	0.350
1145 A/B	AVG	0.365	0.420	0.352	0.355	0.340
	1	0.370	0.460	0.375	0.375	0.360
	2	0.475	0.490	0.360	0.375	0.350
1146 A/B	AVG	0.422	0.475	0.367	0.375	0.355
	1	0.440	0.400	0.480	0.405	0.410
	2	0.445	0.445	0.425	0.410	0.425
1147 A/B	AVG	0.442	0.422	0.452	0.407	0.417
	1	0.410	0.440	0.410	0.355	0.430
	2	0.475	0.435	0.415	0.345	0.395
1710 A/B	AVG	0.422	0.437	0.412	0.350	0.412
	1	-	0.395	0.335	0.330	0.340
	2	-	0.340	0.355	0.340	0.360
1711 A/B	AVG	-	0.367	0.345	0.335	0.350
	1	-	0.400	0.355	0.325	0.325
	2	-	0.390	0.330	0.330	0.330
	AVG	-	0.395	0.342	0.327	0.327

- \* The lubricity of the above fuels was tested on the Furey B.O.C. rig at the onset of the program (0 month interval). The remainder of lubricity testing was performed on the Interav B.O.C. rig.
- \* 84-POSF-1710 and 84-POSF-1711 were not introduced to the program until after the 0 month interval.

TABLE B-8. DRUM CONDUCTIVITY TEST RESULTS

FUEL CODE	0 MONTH TEMP, °F	COND, CU	15 MONTH TEMP, °F	COND, CU
0711	63	210/210 (200)*	69	268/272 (265)
0712	80	275/275 (295)	69	171/169 (270)
0713	83	200/200	69	80/77
0714	61	18/19	69	-----
0715	60	3/3	69	-----
0716	64	160/160	69	1013/1016
0717	82	230/230	69	975/979
0718	76	280/280	9	219/219
0719	65	225/220	69	230/230
0720	60	240/240	69	163/165
0721	52	220/220	69	163/165
0722	77	260/240	69	150/148
0723	64	260/260	69	130/133
0724	58	240/240	69	136/138
0725	83	310/300	69	130/135
0726	69	280/270	69	134/138
0727	77	295/295	69	136/140
0728	66	300/290	69	190/195
0729	66	280/280	69	128/133
0730	90	240/260	69	953/958
0761	78	260/270	69	196/202
1064				

\* ( ) -SFTLA TESTS

TABLE B-9. TEST SAMPLE CONDUCTIVITY TEST RESULTS

FUEL CODE	0 MONTH		9 MONTH		15 MONTH	
	TEMP, °F	COND, CU	TEMP, °F	COND, CU	TEMP, °F	COND, CU
0714	61	18/19				
1130 A/B			70	60/58	69	33/34
1131 A/B			70	3/3	69	3/3
0716 A/B	64	160/160	70		69	1013/1016
1132 A/B			70	290/292	69	114/116
1133 A/B			70	320/315	69	273/269
1134 A/B			70	290/290	69	146/147
1135 A/B			70	143/146	69	106/108
0717	82	230/230	70		69	975/979
1136 A/B			70	93/91	69	80/80
1137 A/B			70	130/128	69	105/108
1138 A/B			70	141/143	69	115/118
0718	76	280/280	70		69	219/219
1139 A/B			70	92/86	69	70/75
1140 A/B			70	89/91	69	97/66
0718 A/B			70	40/40	69	142/141
0718 C/D			70	69/65	69	140/140
0719	65	225/220	70		69	230/230
1141 A/B			70	76/74	69	68/71
1142 A/B			70	180/170	69	168/173
1143 A/B			70	43/43	69	28/30
1144 A/B			70	60/59	69	90/90
0720	60	240/240	70		69	163/165
1145 A/B			70	90/89	69	102/106
1146 A/B			70	67/68	69	107/107
1147 A/B			70	160/159	69	138/138
1148 A/B			70	67/69	69	69/68
0722	77	260/240	70		69	150/148
0722 A/B			70	73/72	69	30/35
0722 C/D			70	93/92	69	20/21
1710 A/B			70	27/26	69	40/42
1711 A/B			70	19/19	69	27/30
0723	64	260/260	70		69	130/133
0723 A/B			70	112/110	69	123/120
1149 A/B			70	106/104	69	100/99
0724	58	240/240	70		69	136/138
0724 A/D			70	70/71	69	108/109
1150 A/B			70	95/94	69	129/123
0725	83	310/300	70		69	130/135
0725			70	104/104	69	173/172
1151 A/B			70	95/94	69	120/120

TABLE B-9. TEST SAMPLE CONDUCTIVITY TEST RESULTS (CON'T)

FUEL CODE	0 MONTH TEMP, °F	COND, CU	9 MONTH TEMP, °F	COND, CU	15 MONTH TEMP, °F	COND, CU
0726	69	280/270	70		69	134/138
0726 A/B			70	75/74	69	137/141
1152 A/B			70	81/83	69	85/86
0727	77	295/295	70		69	136/136
0727 A/B			70	156/155	69	150/146
1153 A/B			70	50/47	69	42/35
0728	66	300/290	70		69	190/195
0728 A/B			70	150/157	69	180/181
1154 A/B			70	121/123	69	102/102
0729	66	280/280	70		69	128/133
0729 A/B			70	70/71	69	124/125
1155 A/B			70	84/83	69	79/79
0730	90	240/260	70		69	953/953
0730 A/B			70	78/79	69	72/75
1156 A/B			70		69	67/64
0761	78	260/270	70		69	196/202
0761 A/B			70	62/63	69	210/208
0761 C/D			70	73/76	69	73/76
1064						
1064 A/B			70	33/32	69	153/153
1064 C/D			70	98/101	69	150/150

## APPENDIX C TEST RESULTS BY SAMPLE NUMBER

The results for each sample are reported at the zero, three, nine and fifteen month sampling intervals. Samples selected for lubricity evaluation were also tested at six months. Two samples containing A09 antioxidant, 1710 and 1711, were added at the 3-month test time, so that 18-month data is included to complete the actual 15-month program. The data is reported as follows:

JFTOT - either as Br Pt (Break Point) or P (Pass) at a given temperature. A pass indicated less than three visual code rating less than 25 mm Hg at the given temperature.

Particulates - mg per liter/filtration time in minutes

Existent Gum - mg per 100 ml

Water Separation - index

Lubricity - wear scar diameter, mm

Peroxides - parts per million

Electrical Conductivity - picosiemens per meter

83-POSF-0711  
 55 Gallon Drum  
 $N_2$  Blanket, Outdoor Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A02	8.4 LB/1000 BBL
C11	8 LB/1000 BBL

TESTING RESULTS

TEST	0 MONTH		3 MONTH		9 MONTH	
	JFTOT	327°C Br Pt	JFTOT	327°C Br Pt	JFTOT	327°C Br Pt
Particulates	0.1/5	0.1/5	0.0/6	0.0/6	0 @ 310	0 @ 310
Existent Gum	0.4	0.4	2.0	2.0	0.2/5	0.2/5
Water Separation	74	74	83	83	0.2	0.2
*Lubricity	.35/.40/.37	.35/.40/.37	.335/.35/.3425	.335/.35/.3425	.33/.34/.335	.33/.34/.335
Peroxides	0.0	0.0	0.0	0.0	0.0	0.0
Electrical Conductivity	200	200	180	180	290	290
FSII	0.13	0.12	0.12	0.12	0.13	0.13

\*6 month Lubricity .335/.33/.3425

83-POSF-0712  
 55 Gallon Drum  
 N<sub>2</sub> Blanket, Outdoor Storage

FSII  
 Conductivity      0.10 - 0.15 volume %  
 A02      1 ppm (ASA - 3 and Stadis - 450)  
 C11      8.4 LB/1000 BBL  
               8 LB/1000 BBL

#### TESTING RESULTS

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	320°C Br Pt	JFTOT	P @ 310	0.1/5	0.0/6	0.0/5	320°C Br Pt	0.3/5
Particulates	0.1/5	0.0	0.4	0.4	1.6	1.6	1.0	1.0
Existent Gum	0.0	66	85	70	70	66		
Water Separation	66							
*Lubricity	.38/.375/.377	.34/.365/.3525	.33/.325/.3275	.325/.325/.325				
Peroxides	0.0	0.0	4.0	0.0				
Electrical Conductivity	295	200	300	260/171/169				
FSII	0.12	0.10	0.13	0.13				

\*6 month Lubricity .33/.335/.3325

83-POSF-0718 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage  
 FSII 0.7 - 0.15 volume  
 Conductivity 1 ppm (ASA - 3 and Stadis - 450)  
 A02 8.4 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	6 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P	0	P	0	P	0	P	0
JFTOT	313	°C Br Pt	P @ 310	0	P @ 310	0	P @ 310	0
Particulates	0.1/4		0.2/6	0	0.0/5	0	0.1/5	0
Existent Gum	0.2		0.0		0.4		0.0	
Water Separation	90		99		88		93	
*Lubricity	.575/.635/.605		.43/.47/.45		.39/.46/.425		.750/.520/.675/.648	
Peroxides	0.0		0.0		0.0		0.0	
Electrical Conductivity	270				135/132		142/141	
FSII	0.12							

\*6 month Lubricity .47/.47/.47

83-POSF-1718 (C&D)  
5 Gallon Cans  
Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A02	8.4 LB/10000

## TESTING RESULTS (C&D)

<u>TEST</u>	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>	<u>15 MONTH</u>
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.1/4	0.1/6	0.1/5	0.1/5
Existent Gum	0.0	0.0	0.4	0.0
Water Separation	90	99	93	93
*Lubricity	.815/.805/.81	.555/.555/.5525	.605/.70/.6525	.750/.520/.675/.648
Peroxides	0.0	0.0	0.32	0.0
Electrical Conductivity			200/200	142/141

\*6 month Lubricity .525/.545/.535

83-POSF-0722 (A&B)  
 5 Gallon Cans  
 Oven Storage

FSII 0.10 - 0.15 volume %  
 Conductivity 1 ppm (ASA - 3 and Stadis - 450)  
 CII 4 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH		
	JFTOT	320 °C Br Pt	Particulates	0.2/4	0.2/6	P @ 320	0.2/5	P @ 280	0.5/3
Particulates		0.2/4		0.2	0.0	0.0	0.4	0.0	0.0
Existent Gum		0.2		78	88	95	95	72	72
Water Separation		78							
*Lubricity	.405/.136/.382			.435/.44/.4375		.38/.345/.362			
Peroxides		0.0		0.0		0.64/0.80			
Electrical Conductivity		290				220/214			
FSII		0.14							

\*6 month Lubricity 0.32/.335/.3275

83-POSF-0722 (C&D)  
5 Gallon Cans  
Oven Storage

FSII            0.10 - 0.15 volume %  
Conductivity    1 ppm (ASA - 3 and Stadis - 450)  
C11            4 LB/1000 BBL

TESTING RESULTS (C&D)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 320	P @ 320	P @ 320	P @ 280
Particulates	0.1/4	0.2/6	0.2/5	1/5
Existent Gum	0.0	0.6	0.0	3.4
Water Separation	90	85	94	56
*Lubricity	.49/.34/.41	.44/.415/.4275	.34/.35/.345	(C) 538/460
Peroxides	0.0	0.0	2.2/2.4	(D) 538/460
Electrical Conductivity			170/172	20/21
FSII				

\*6 month Lubricity .31/.335/.3225

83-POSF-0723 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
CI1 1 ppm (ASA - 3 and Stadis - 450)  
CI1 4 LB/1000 BBL  
A02 6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @	320	P @	320	P @	320	P @	320
JFTOT	0.1/4	0.2/6	0.1/4	0.2/5	0.1/4	0.2/5	0.1/4	0.2/5
Particulates	0.4	0.6	0.4	0.6	0.4	0.6	0.4	0.6
Existent Gum								
Lubricity								
Water Separation	74	96	100	91	100	91	100	91
Peroxides	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Electrical Conductivity	(260 Drum)		220/230		123/120			
FSII								

83-POSF-0724 (A&B)  
5 Gallon Cans  
Oven Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
CI1	4 LB/1000 BBL
A03	6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 320	P @ 300	P @ 320
Particulates	0.1/4	0.2/6	0.3/5	0.2/5
Existent Gum	0.0	0.4	0.0	0.6
Water Separation	79	96	96	93
Lubricity				
Peroxides	0.0	0.0	0.0	0.415
Electrical Conductivity	(240 Drum)	250/260	108/109	
FSII				

83-POSF-0725 (A&B)  
 5 Gallon Cans  
 Oven Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
CI1	4 LB/1000 BBL
A04	6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	TESTING RESULTS (A&B)		
	0 MONTH	3 MONTH	9 MONTH
JFTOT	P @ 320	P @ 320	P @ 320
Particulates	0.2/4	0.2/6	0.2/5
Existent Gum	0.0	0.4	1.2
Water Separation	85	91	90
Lubricity			
Peroxides	0.0	0.0	0.520
Electrical Conductivity	(305 Drum)	220/220	173/172
FSII			

83-POSF-0726 (A&B)  
5 Gallon Cans  
Oven Storage

FSII                    0.10 - 0.15 volume %  
Conductivity        1 ppm (ASA - 3 and Staddis - 450)  
C11                    4 LB/1000 BBL  
A05                    6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 320	P @ 300	P @ 320	P @ 320
Particulates	0.2/4	0.2/6	0.1/5	0.1/4
Existent Gum	0.0	0.2	0.6	0.6
Water Separation	74	88	92	84
Lubricity				
Peroxides	0.0	0.0	0.0	0.0
Electrical Conductivity	(275 Drum)	240/240	137/141	
FSII				

83-P0SF-0727 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
C11 1 ppm (ASA - 3 and Staddis - 450)  
A06 4 LB/1000 BBL  
6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @ 300	P @ 320	P @ 320	P @ 320	0.2/6	0.2/6	0.1/4	0.1/4
JFTOT	0.1/4	0.2/6	0.2/6	0.2/6	0.6	0.6	0.6	0.6
Particulates	0.6	0.0	0.0	0.0	84	84	84	84
Existent Gum	78	90	90	90				
Water Separation								
Water Separation								
Lubricity								
Peroxides	0.0	0.0	0.0	0.0				
Electrical Conductivity	(295 Drum)	0.32	0.32	0.32	260/260	260/260	0.0	0.0
FSII								

83-POSF-0728 (A&B)  
 5 Gallon Cans  
 Oven Storage  
  
 FSI  
 Conductivity 0.10 - 0.15 volume %  
 1 ppm (ASA - 3 and Stadis - 450)  
 C11 4 LB/1000 BBL  
 A07 6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @ 300	P @ 300	P @ 300	P @ 300	P @ 320	P @ 320	P @ 320	P @ 320
JFTOT	0.1/4	0.2/5	0.1/5	0.2/5	0.4	0.2/5	0.4	0.2/5
Particulates	0.0	0.8	0.2	0.8	72	93	72	93
Existent Gum	77	99	93	99				
Water Separation								
Lubricity								
Peroxides	0.0	0.0	0.0	0.0	1.442	1.442	1.442	1.442
Electrical Conductivity	(300 Drum)	(300 Drum)	(300 Drum)	(300 Drum)	355/360	355/360	355/360	355/360
FSII								

83-POSF-0729 (A&B)  
5 Gallon Cans  
Over Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Staddis - 450)
C11	4 LB/1000 BBL
A08	6 LB/1000 BBL

#### TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @	320	P @	320	P @	320	P @	320
JFTOT	0.1/4		0.1/5		0.2/5		0.2/5	
Particulates	0.6		0.2		0.0		0.4	
Existent Gum	82		96		96		81	
Water Separation								
Lubricity								
Peroxides	0.0		0.0		0.0		5.56	
Electrical Conductivity	(280 Drum)		260/260		124/125		124/125	
FSII								

83-POSF-0730 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
CII 1 ppm (ASA - 3 and Stadis - 450)  
A01 4 LB/1000 BBL  
6 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 300	P @ 300	P @ 300	P @ 300
Particulates	0.1/5	0.1/6	0.2/6	0.1/4
Existent Gum	0.6	0.4	0.4	0.2
Water Separation	63	93	89	93
Lubricity				
Peroxides	0.0	0.0	0.0/0.08	0.0
Electrical Conductivity	(250 Drum)	85/89	72/75	
FSII				

83-POSF-0761 (A&E)  
 5 Gallon Cans  
 Room Temperature Storage

FSII Conductivity 0.10 - 0.15 volume %  
 1 ppm (ASA - 3 and Stadis - 450)

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.1/5	0.1/6	0.2/5	0.1/4
Exsistent Gum	0.6	0.4	0.6	0.6
Water Separation	92	97	92	99
*Lubricity	0.815/.76/.787	0.53/.52/.525	.460/.445/.4525	1.105/.605/.435/.715
Peroxides	0.0	0.0	0.08	0.0
Electrical Conductivity			183/180	216/208
FSII				

\*6 month Lubricity .565/.65/.6075

83-POSF-0761 (C&D)  
 5 Gallon Cans  
 Room Temperature Storage

FSII 0.10 - 0.15 volume %  
 Conductivity 1 ppm (ASA - 3 and Stadis - 450)

TESTING RESULTS (C&D)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	JFTOT	Particulates	Existent Gum	Water Separation	*Lubricity	Peroxides	Electrical Conductivity	FSII
P @ 310	0.1/5	0.4	89	.525/.48/.502	.555/.59/.5725	0.0	.44/.49/.465	P @ 310
0.3/6	0.4	99	0.0	0.0	0.0	0.0	.44/.49/.465	0.2/5
0.0	0.0	89	.5725	0.0	0.0	0.0	.910/.615/.435/.653	1.0
						170/170		90
								112/110

83-POSF-1064 (Petroleum) (A&B)  
5 Gallon Cans  
Oven Storage

SPECIFICATION

TESTING RESULTS (A&B)

<u>TEST</u>	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>	<u>15 MONTH</u>
JFTOT	P @ 300	P @ 300	P @ 320	P @ 320
Particulates	0.2/5	0.2/5	0.2/5	0.1/4
Existent Gum	0.2	0.0	0.0	0.2
Water Separation	67	98	97	97
Lubricity				
Peroxides		0.0	0.08	1.151
Electrical Conductivity		130/132	130/132	153/153
FSII				

83-POSF-1064 (Petroleum) (C&D)  
5 Gallon Cans  
Room Temperature Storage

SPECIFICATION

TESTING RESULTS (C&D)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.1/5	0.2/6	0.1/5	0.1/4
Existent Gum	0.0	0.0	0.0	0.0
Water Separation	85	79	86	89
*Lubricity	.47/.435/.452	.345/.345/.345	.355/.335/.345	.320/.315/.3175
Peroxides	0.074	0	0.24	0
Electrical Conductivity			485/490	150/150
FSII				

83-POSF-1130 (A&B)  
5 Gallon Cans  
Room Temperature Storage

FSII	0.10 - 0.15 volume %
A02	8.4 LB/1000 BBL
C11	8 LB/1000 BBL

#### TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 320	P @ 290	P @ 310	P @ 290
Particulates	0.2/5	0.1/5	0.3/8	0.1/5
Existent Gum	0.6	0.0	1.0	0.6
Water Separation	75	81	82	92
*Lubricity				
Peroxides				
Electrical Conductivity			3/0	33/34
FSII				

83-POSF-1131 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
A01	8.4 LB/1000 BBL
C11	8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 290	P @ 310	P @ 310
Particulates	0.1/4	0.1/4	0.1/5	0.1/5
Existent Gum	0.0	0.4	0.0	0.4
Water Separation	88	86	88	95
Lubricity				
Peroxides				
Electrical Conductivity				
FSII		0/3	3/3	

83-POSF-1132 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

	FSII	Conductivity	0.10 - 0.15 volume %
A01	1 ppm (ASA - 3 and Stadis - 450)	8.4 LB/1000 BBL	
C11	8 LB/1000 BBL		

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 290	P @ 310	P @ 290
Particulates	0.2/4	0.2/6	0.4/6	0.2/5
Existent Gum	0.4	0.4	0.0	0.0
Water Separation	72	74	56	86
*Lubricity	.31/.305/.307	.31/.305/.3075	.32/.31/.315	.300/.310/.305
Peroxides				
Electrical Conductivity				
FSII		160/160		114/116

\*6 month Lubricity .345/.35/.3475

83-POSF-1133 (A&B)  
5 Gallon Cans  
Room Temperature Storage

FSI  
Conductivity 0.10 - 0.15 volume %  
A01 1 ppm (ASA - 3 and Stadis - 450)  
C12 8.4 LB/1000 BBL  
8 LB/1000 BBL

TESTING RESULTS (A&B)

<u>TEST</u>	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>	<u>15 MONTH</u>
JFTOT	P @ 300	P @ 290	P @ 310	P @ 310
Particulates	0.3/5	0.1/5	0.2/5	0.3/5
Existent Gum	0.0	0.6	0.0	0.2
Water Separation	81	90	65	73
*Lubricity				
Peroxides				
Electrical Conductivity			220/220	273/269

83-POSF-1134 (A&B)  
5 Gallon Cans  
Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A01	8.4 LB/1000 BBL
C13	8 LB/1000 BBL

## TESTING RESULTS (A&B)

83-POSF-1135 (A&B)  
5 Gallon Cans  
Room Temperature Storage

FSII 0.10 - 0.15 volume %  
Conductivity 1 ppm (ASA - 3 and Stadis - 450)  
A01 8.4 LB/1000 BBL  
C14 8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
UFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.3/4	0.1/6	0.3/6	0.2/4
Existent Gum	0.6	0.0	0.0	0.0
Water Separation	71	61	58	67
Lubricity				
Peroxides				
Electrical Conductivity				
FSII		135/140	106/108	

83-POSF-1136 (A&B)  
5 Gallon Cans  
Room Temperature Storage

FSII Conductivity 0.10 - 0.15 volume %  
A01 1 ppm (ASA - 3 and Stadis - 450)  
C11 8.4 LB/1000 BBL  
Metal Deactivator 8 LB/1000 BBL  
                  2 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		6 MONTH		9 MONTH		15 MONTH	
	P @ 320	JFTOT	P @ 310	Particulates	P @ 310	Existent Gum	0.3/6	P @ 310
Particulates	0.2/4	0.1/5	0.1/5	0.6	0.4	1.2	0.2/4	0.2/4
Existent Gum				0.6	0.4	1.2	0.6	0.6
Water Separation				86	77	60		81
Lubricity								
Peroxides								
Electrical Conductivity								
FSII								

83-POSF-1137 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 Rpm (ASA - 3 and Stadis - 450)
A01	8.4 LB/1000 BBL
C12	3 LB/1000 BL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.5/4	0.1/6	0.2/6	0.3/4
Existent Gum	0.4	0.0	0.2	0.4
Water Separation	91	95	79	88
*Lubricity	.38/.47/.425	.43/.56/.495	.365/.365/.365	.395/.365/.38
Peroxides				
Electrical Conductivity				
FSII		170/169	105/108	

\*6 month Lubricity .46/.375/.4175

83-POSF-1138 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

	FSI I	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)	
A01	8.4 LB/1000 BBL	
C11	8 LB/1000 BBL	
JFA-5	4 LB/1000 BBL	

TESTING RESULTS (B&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @ 310	P @ 300	P @ 310	P @ 300	P @ 310	P @ 300	P @ 310	P @ 300
JFTOT	0.1/3	0.1/7	0.2/6	0.2/5	0.2/6	0.2/5	0.2/6	0.2/5
Particulates	0.8	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Existent Gum	39	57	41	56	41	56	41	56
Water Separation								
Lubricity								
Peroxides								
Electrical Conductivity								
FSII								
	96/99	115/118						

83-POSF-1139 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A02	8.4 LB/1000 BBL
C11	8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.1/3	0.1/6	0.2/5	0.3/4
Existent Gum	0.8	0.0	0.6	0.0
Water Separation	87	88	67	95
*Lubricity	.27/.32/.295	.31/.305/.3075	.325/.31/.3175	.320/.305/.3125
Peroxides				
Electrical Conductivity				
FSII		171/169	70/75	

\*6 month Lubricity .345/.305/.325

83-POSF-1140 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII  
 Conductivity 0.10 - 0.15 volume %  
 1 ppm (ASA - 3 and Stadis - 450)  
 A02 8.4 LB/1000 BBL  
 C12 8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.4/5	0.2/6	0.1/5	0.1/4
Existent Gum	0.0	0.0	0.0	0.0
Water Separation	78	87	81	89
*Lubricity	.37/.35/.36	.335/.34/.3375	.335/.335/.335	.315/.310/.3125
Peroxides				
Electrical Conductivity			170/170	97/96
FSII				

83-POSF-1141 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A02	8.4 LB/1000 BBL
C13	8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @ 310	P @ 310	P @ 310	P @ 310	P @ 310	P @ 310	P @ 290	P @ 290
JFTOT	0.3/4	0.2/6	0.2/5	0.2/4	0.0	0.0	0.2/4	0.0
Particulates	0.6	0.4	0.0	0.0	63	76	76	76
Existent Gum	73	81	63	63				
Water Separation								
*Lubricity	.375/.405/.39	.44/.455/.4475	.345/.395/.37	.345/.395/.37				
Peroxides								
Electrical Conductivity								
FSII								

83-POSF-1142 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII 0.10 - 0.15 volume %  
 Conductivity 1 ppm (ASA - 3 and Stadis - 450)  
 A02 8.4 LB/1000 BBL  
 C14 8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.2/4	0.1/6	0.0/5	0.2/4
Existent Gum	0.6	0.8	0.8	0.8
Water Separation	66	88	73	74
*Lubricity	.37/.335/.352	.44/.45/.445	.345/.335/.34	.325/.320/.3225
Peroxides				
Electrical Conductivity			157/157	168/173

\*6 month Lubricity .36/.335/.3475

83-POSF-1143 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII  
 Conductivity      0.10 - 0.15 volume %  
 A02              1 ppm (ASA - 3 and Stadis - 450)  
 8.4 LB/1000 BBL  
 CII              8 LB/1000 BBL  
 Metal Deactivator      2 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.2/4	0.3/6	0.2/6	0.3/5
Existent Gum	0.0	0.0	0.0	0.2
Water Separation	85	91	57	67
Lubricity				
Peroxides				
Electrical Conductivity				
FSII			55/60	28/30

83-POSF-1144 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A02	8.4 LB/1000 BBL
C11	3 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	TEST		
	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>
JFTOT	P @ 310	P @ 310	P @ 310
Particulates	0.3/5	0.3/6	0.1/6
Existent Gum	0.0	0.4	0.4
Water Separation	83	92	72
*Lubricity	.365/.365/.365	.43/.41/.42	.35/.36/.355
Peroxides			.330/.350/.340
Electrical Conductivity			161/160
FSII			90/90

\*6 month Lubricity .345/.36/.3525

83-POSF-1145 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII  
 Conductivity 0.10 - 0.15 volume %  
 A02 1 ppm (ASA - 3 and Stadis - 450)  
 C12 8.4 LB/1000 BBL  
 3 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	P @ 310	P @ 310	P @ 310	P @ 310	0.2/6	0.2/6	0.3/4	0.3/4
JFTOT	0.3/5	0.0	0.4	0.4	0.4	0.4	0.2	0.2
Particulates								
Existent Gum								
Water Separation	84		93		77		90	
*Lubricity	.37/.475/.422		.46/.49/.475		0.375/.375/.375		.360/.350/.355	
Peroxides								
Electrical Conductivity								
FSII					190/180		102/106	

\*6 month Lubricity .375/.36/.3675

83-POSF-1146 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII            0.10 - 0.15 volume %  
 Conductivity    1 ppm (ASA - 3 and Staddis - 450)  
 A02            8.4 LB/1000 BBL  
 C13            3 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.2/4	0.1/6	0.1/5	0.1/5
Existent Gum	0.4	0.0	0.0	0.4
Water Separation	83	81	95	86
*Lubricity	.44/.445/.442	.40/.445/.4225	.405/.41/.4075	.410/.425/.4175
Peroxides				
Electrical Conductivity			140/140	107/107
FSII				

83-POSF-1147 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
A02	8.4 LB/1000 BBL
C14	3 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	15 MONTH		
	0 MONTH	3 MONTH	9 MONTH
JFTOT	P @ 310	P @ 310	P @ 310
Particulates	0.3/4	0.0/6	0.1/6
Existent Gum	0.4	0.2	0.4
Water Separation	90	90	77
*Lubricity	.41/.475/.442	.44/.435/.4375	.355/.345/.35
Peroxides			.430/.395/.4125
Electrical Conductivity			160/160
FSII			138/138

\*6 month Lubricity .410/.415/.4125

83-POSF-1148 (A&B)  
 5 Gallon Cans  
 Room Temperature Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Staddis - 450)
A02	8.4 LB/1000 BBL
CI1	8 LB/1000 BBL
JFA-5	4 LB/1000 BBL

TESTING RESULTS (A&B)

<u>TEST</u>	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>	<u>15 MONTH</u>
JFTOT	P @ 310	P @ 310	P @ 310	P @ 310
Particulates	0.1/4	0.2/7	0.1/6	0.3/5
Existent Gum	1.0	0.0	0.2	0.0
Water Separation	49	55	40	61
Lubricity				
Peroxides				
Electrical Conductivity			220/220	69/68
FSII				

83-POSF-1149 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity      0.10 - 0.15 volume %  
C11      1 ppm (ASA - 3 and Staddis - 450)  
A02      4 LB/1000 BBL  
              16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 310	P @ 320	P @ 320	P @ 320
Particulates	17.2/4	0.1/5	0.2/5	0.3/5
Existent Gum	0.0	0.2	0.4	0.0
Water Separation	86	94	87	85
Lubricity				
Peroxides	0.0	0.0	0.0	0.0
Electrical Conductivity			190/189	100/99
FSII				

83-POSF-1150 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
C11 1 ppm (ASA - 3 and Staddis - 450)  
A03 4 LB/1000 BBL  
16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH		15 MONTH	
	JFTOT	P @ 320	P @ 320	P @ 320	P @ 300	P @ 320	P @ 300	P @ 300
Particulates	0.1/4	0.2/5	0.2/5	0.3/5	0.2/5	0.4	0.2/5	0.2/5
Existent Gum	0.2	0.0	0.0	0.4	0.0	98	0.0	0.0
Water Separation	84	94	94	96	96	98	96	96
Lubricity								
Peroxides		0.0	0.0	0.0	0.0	0.0	0.0	0.0
Electrical Conductivity			250/240			129/123		
FSII								

83-POSF-1151 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
CI1 1 ppm (ASA - 3 and Stadis - 450)  
A04 4 LB/1000 BBL  
16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH	
	P @ 320	P @ 300	0.2/5	0.0/6	0.2	0.5/5
JFTOT	0.2/5	0.0/6	0.0	0.2	0.6	0.6
Particulates	92	96	96	96	90	90
Existent Gum						
Water Separation						
Lubricity						
Peroxides						
Electrical Conductivity	0.0	0.0	0.0	0.0	0.0	0.0
FSII						

83-POSF-1152 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
CI1 1 ppm (ASA - 3 and Stadis - 450)  
A05 4 LB/1000 BBL  
16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH
JFTOT	P @ 310	P @ 320	P @ 320
Particulates	0.1/4	0.0/6	0.3/6
Existent Gum	0.0	0.0	0.8
Water Separation	92	90	96
Lubricity			
Peroxides	0.0	0.0	0.0
Electrical Conductivity			230/230
FSII			

83-POSF-1153 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
C11 1 ppm (ASA - 3 and Stadis - 450)  
A06 4 LB/1000 BBL  
16.8 LB/1000 BBL

TESTING RESULTS (A&B)

<u>TEST</u>	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>
JFTOT	P @ 320	P @ 320	P @ 320
Particulates	0.2/4	0.2/6	0.2/6
Existent Gum	0.0	0.0	0.0
Water Separation	82	75	86
Lubricity			
Peroxides			
Electrical Conductivity	0.0	0.0	0.96
FSII			59/60

83-POSF-1154 (A&B)  
5 Gallon Cans  
Oven Storage

FSII  
Conductivity 0.10 - 0.15 volume %  
CII 1 ppm (ASA - 3 and Stadis - 450)  
A07 4 LB/1000 BBL  
16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH
JFTOT	P @ 300	P @ 300	P @ 320
Particulates	0.2/4	0.1/4	0.4/5
Existent Gum	0.4	0.8	0.2
Water Separation	89	86	94
Lubricity			
Peroxides			
Electrical Conductivity	0.0	0.0	0.48
FSII			280/280

83-POSF-1155 (A&B)  
5 Gallon Cans  
Oven Storage

FSII Conductivity 0.10 - 0.15 volume %  
C11 1 ppm (ASA - 3 and Stadis - 450)  
A08 4 LB/1000 BBL  
16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH
JFTOT	P @ 300	P @ 320	P @ 320
Particulates	0.2/4	0.1/6	0.1/6
Existent Gum	0.4	0.4	0.6
Water Separation	89	93	92
Lubricity			
Peroxides	0.0	0.0	3.4/3.0
Electrical Conductivity			170/170
FSII			

83-POSF-1156 (A&B)  
5 Gallon Cans  
Oven Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
CII	4 LB/1000 BBL
A01	16.8 LB/1000 BBL

#### TESTING RESULTS (A&B)

TEST	0 MONTH		3 MONTH		9 MONTH	
	JFTOT	P @ 300	JFTOT	P @ 300	Particulates	P @ 300
Particulates	0.2/4	0.1/5	0.0	0.8	0.3/5	0.4
Existent Gum	0.0	76	88	77	77	77
Water Separation						
Lubricity						
Peroxides						
Electrical Conductivity	0.0	0.0	0.32	0.0	90/88	90/88
FSII						

84-POSF-1710 (A&B)  
5 Gallon Cans  
Over Storage

FSII                    0.10 - 0.15 volume %  
Conductivity        1 ppm (ASA - 3 and Stadis - 450)  
C11                    4 LB/1000 BBL  
A09                    6 LB/1000 BBL

TESTING RESULTS (A&B)

<u>TEST</u>	<u>0 MONTH</u>	<u>3 MONTH</u>	<u>9 MONTH</u>	<u>15 MONTH</u>
JFTOT	No Data	P @ 320	P @ 320	P A 320
Particulates		0.2/5	0.2/6	0.14
Existent Gum		0.6	0.8	0.0
Water Separation		88	81	89
Lubricity	.395/.34/.3675	.33/.34/.335	.34/.36/.35	
Peroxides	0.0	0.64	1.553/1.740	
Electrical Conductivity	110	205/200	40/42	
FSII				

\*6 month Lubricity .335/.355/.345

84-POSF-1711 (A&B)  
 5 Gallon Cans  
 Oven Storage

FSII	0.10 - 0.15 volume %
Conductivity	1 ppm (ASA - 3 and Stadis - 450)
C11	4 LB/1000 BBL
A09	16.8 LB/1000 BBL

TESTING RESULTS (A&B)

TEST	0 MONTH	3 MONTH	9 MONTH	15 MONTH
JFTOT	P @ 320	P @ 320	P @ 320	P @ 320
Particulates	0.4/4	0.3/5	0.3/5	0.3/5
Existent Gum	0.4	0.6	0.6	0.8
Water Separation	65	57	57	57
Lubricity	.40/.39/.395	.325/.33/.3275	.325/.330/.3275	.325/.330/.3275
Peroxides		0.880		1.783/2.830
Electrical Conductivity	95	115/114	27/30	27/30
FSII				

\*6 month Lubricity .355/.330/.342

## APPENDIX D TOTAL INSOLUBLES TEST PROCEDURES

### FUEL STORAGE STABILITY

Objective: To determine the amount of total insolubles which result from stressing fuel samples at a temperature of 43°C.

Test Schedule: Two tests and one blank for each fuel sample at each test period.

#### Test Periods

Time at 43°C	Equivalent Time at Ambient
0 weeks	0 years
13 weeks	1 year
26 weeks	2 years
39 weeks	3 years

#### Equipment/Supplies:

1. Analytical Balances (2)
  - a. Mettler Balance (for filter weighing)
  - b. B-5C1000 (J9000/JH0975) Balance
2. Drying oven - either of the blue drying ovens that were located in the labs of Bldg 59C.
3. Stressing oven - the yellow oven for long term fuel storage which used to be in Bldg 59C lab annex. Oven should be capable of  $43 \pm 1^\circ\text{C}$  for extended periods of time.
4. Petri dishes, glass, to hold 47 mm filter, with lid.
5. Forceps, flat-bladed, non-pointed tip.
6. Solvents, HPLC methanol and toluene in a filtered wash bottle.
7. Iso-octane, HPLC in filtered wash bottle.
8. Filters, one test and one control filter for each 400 ml fuel sample, 47 mm diameter, nominal pore size 0.8  $\mu\text{m}$ .
9. Erlenmeyer flasks, 500 ml, 12 for each fuel to be tested, with caps and teflon liners.
10. Dishwasher
11. Aluminum foil
12. Carbide etching pen
13. Laboratory filtration apparatus (See Figure 1)

Preparation of Sample Containers:

- Initially number each 500 ml Erlenmeyer flask (screw top, borosilicate) with a carbide etching pen.
- Rinse flasks, caps, and liners with an equal volume mixture of methanol and toluene from a filtered wash bottle.
- Put flasks into dishwasher for normal cycle.
- Repeat rinse cycle on dishwasher (note: dishwasher uses distilled water).
- Place flasks, caps and liners in drying oven at  $110^{\circ} \pm 10^{\circ}\text{C}$  for at least 8 hours (caps, liners, flasks need not be assembled.)
- Place teflon lined caps loosely on containers and cool overnight.
- Cover flasks or place in cabinet to avoid airborne dust.

Note: Technique is very important in this test since the amount of sediment and gum may be less than 1 mg. All containers should be wiped off with a clean dry towel before weighing to remove any airborne dust.

Procedure for Testing for Insolubles:

1. Weigh the cleaned, cool, marked 500 ml Erlenmeyer flasks (without lids) making sure to use proper technique in order to avoid fingerprints and oil on the flasks. A "B-5C1000 (J9000/JH0975) Balance (1000 grams max.) should be used for this weighing. Each fuel sample to be tested will require twelve different flasks; one for the test, one for the duplicate and one for the blank, for each of four testing periods.
2. Fill each test and duplicate flask with 400 ml (four hundred) of each fuel to be tested. Blanks should be prepared as in Step 7. Securely tighten the teflon-lined caps onto the containers and wrap flasks with aluminum foil. Place containers in the stressing oven at a temperature of  $43^{\circ}\text{C} (\pm 1^{\circ}\text{C})$ . Leave samples in oven with minimal disturbances until times indicated in the test schedule. (The samples to be tested at 0 weeks will not be stressed at all).
3. At the end of each testing period, remove samples from oven and allow to cool overnight.
  - a. Two 47 mm diameter filters of nominal pore size  $0.8 \mu\text{m}$  are required for each flask. One filter is a test filter and one is a control filter. Each of these filters should be placed in a clean, dry Petri dish (with lid) and appropriately marked.

b. After having used forceps to lay each filter in a clean Petri dish, place dish and filter, with lid slightly ajar in drying oven at 11 °C for 30 minutes to remove water adsorbed from the atmosphere onto the filter. With the lid still slightly ajar, remove dishes from oven and allow filters to come to equilibrium with the atmosphere (about 30 more minutes). Carefully weigh each filter on a mettler balance which will weigh to the nearest 0.1 mg without interpolation and return weighed filters to respective Petri dishes. Record weights.

4. The laboratory filtration apparatus is shown in Figure 1 below:

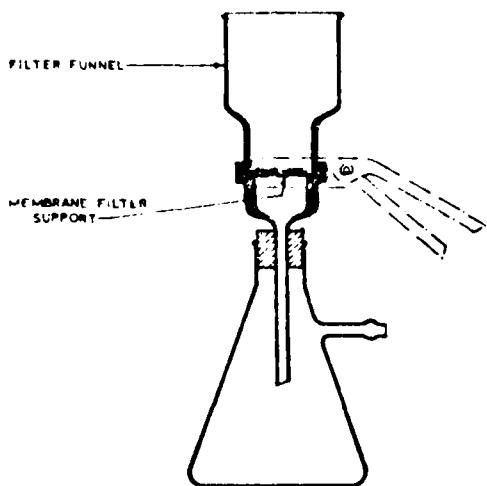


Figure 1 Apparatus for Determining Total Contaminant Bottle Samples

- a. Using a clean forcep, assemble the filtering apparatus with the control filter under the test filter (both already weighed).
- b. Wet both filters with approximately 100 ml of the filtered flushing fluid. (Note: it is not necessary to use an exact amount of iso-octane for this rinsing of filters. It is, however, important to use the same amount of solvent for each determination. Therefore, if a total volume of 400 ml (exactly) - 100 ml for this rinse and 300 ml for step 4e - is used, one can control the amount of solvent for each determination.
- c. Shake the sample container vigorously for about 30 seconds. Remove the cap and any external contaminant that may be present in the threads on the sample container by washing with filtered flushing fluid. Ensure that none of the washings enter the container.

d. Pour some of the sample into the filter funnel. Apply vacuum to the flask and maintain a liquid head in the funnel until completion of filtration by suitable transference of the remainder of the sample, agitating the sample container before each addition. Disconnect the vacuum and record the volume of filtered sample.

e. Use 250 to 300 ml of filtered flushing fluid in this and the succeeding paragraph. Wash the sample container with four 50 ml quantities of filtered flushing fluid to complete transference of the contaminant to the filter.

f. Wash down the inside of the funnel with filtered flushing fluid. With the vacuum applied, carefully remove the clamp and funnel. Wash the periphery of the filter by directing a gentle stream of flushing fluid from the edge to the center, taking great care not to wash any of the contaminant from the surface of the test filter. Maintain vacuum after the final washing only for the few seconds necessary to remove excess fluid from the filter.

g. Using clean forceps, carefully remove the test and control filters from the filter base and place them in a clean covered Petri dish, taking care not to disturb the contaminant on the surface of the filters. Repeat the procedure described in 3b. (May need to allow up to 4 hours for filters to dry in oven).

5. The change in weight of the dry test filter contaminant minus the change in weight of the control filter is equal to the weight of filterable sediments in the fluid.

6. To determine the amount of adherent gum, allow the container to dry in the oven (described in 3b) overnight. After allowing container to cool for at least four hours, weigh flask (without lids or foil) and subtract original weight of container at the start of the test. (Again, make sure not to contaminate flask with fingerprints or oil).

7. Blanks for this test should be treated exactly as the other samples in the test. The same amounts of filtered flushing fluid should be used as in steps 4b, 4e, and 4f. Weight changes should be subtracted from values for adherent gum (whether positive or negative).

8. Total insolubles is the sum of filterable sediment and adherent gum.

9. If any instructions are not clear, or if any changes seem advisable, please contact me before proceeding.

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